

Supporting Information

Synthesis of Primary and Secondary Alkylboronates through Site-Selective C(sp³)-H Activation with Silica-Supported Monophosphine-Ir Catalysts

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Table of Contents

Instrumentation and Chemicals	S1–S2
Experimental Procedure	S2–S3
Ligand Effect	S3–S4
Compounds Characterization	S4–S7
References	S7–S8
NMR Spectra	S9–S32

Instrumentation and Chemicals

Solution NMR spectra were recorded on a Varian Gemini 2000 spectrometer, operating at 300 MHz for ¹H NMR, 75.4 MHz for ¹³C NMR. Chemical shift values for ¹H and ¹³C are reference to Me₄Si and the residual solvent resonances respectively. Chemical shifts are reported in δ ppm. High-resolution mass spectra were recorded on a Thermo Fisher Scientific Exactive, JEOL JMS-T100LP mass spectrometer or JEOL JMS-T100 GC mass spectrometer at the Instrumental Analysis Division, Equipment Management Center, Creative Research Institution, Hokkaido University. TLC analyses were performed on commercial glass plates bearing 0.25-mm layer of Merck Silica gel 60F₂₅₄. Silica gel (Kanto Chemical Co., Silica gel 60 N, spherical, neutral) was used for column chromatography. GLC analyses were conducted on a Shimadzu GC-14B equipped with a flame ionization detector. Melting points were determined on a micro melting point apparatus (Yanaco: MP-500D) using micro cover glass.

All reactions were carried out under nitrogen atmosphere. Materials were obtained from commercial suppliers or prepared according to standard procedures unless otherwise noted. [Ir(OMe)(cod)]₂ was prepared according to the literature.¹ Silica-SMAP² and Silica-TRIP³ were prepared with CARiACT Q-10[®] according to the reported procedure. CARiACT Q-10 (Catalyst grade, 75-150 μm) was purchased from Fuji Silysia Chemical Ltd. and dehydrated by heating at 150 °C for 10 h and stored in a glovebox before use. All solvents for catalytic reactions were

degassed via four freeze–pump–thaw cycles before use. Bis(pinacolato)diboron was purchased from AllyChem Co., Ltd., and recrystallized from pentane before use.

Experimental Procedures

Typical Procedure for the Borylation of 2-Ethylpyridine (1a) with Immobilized Ligands (Table 1, entry 1). In a glove box, Silica-SMAP (0.070 mmol/g, 85.7 mg, 0.006 mmol, 2.0 mol%), bis(pinacolato)diboron (**2**) (76.2 mg, 0.30 mmol), and anhydrous, degassed TBME (1.0 mL) were placed in a 10 mL glass tube containing a magnetic stirring bar. A solution of [Ir(OMe)(cod)]₂ (2.0 mg, 0.003 mmol, 1.0 mol%) in TBME (2.0 mL) and 2-ethylpyridine (**1a**) (96.4 mg, 0.90 mmol) were added successively. The tube was sealed with a screw cap and removed from the glovebox. The reaction mixture was stirred at 25 °C for 12 h, and filtered through a glass pipet equipped with a cotton filter. The solvent was removed under reduced pressure. An internal standard (1,1,2,2-tetrachloroethane) was added to the residue. The yields of the products **3a** and **4a** were determined by ¹H NMR (66% and 21% respectively). The crude material was then purified by Kugelrohr distillation, to give the corresponding product **3a** (45.2 mg, 0.19 mmol in 64% isolated yield).

Typical Procedure for the Borylation of 2-Ethylpyridine (1a) with Soluble Ligands (Table 1, entry 4). In a glove box, bis(pinacolato)diboron (**2**) (76.2 mg, 0.30 mmol) and anhydrous, degassed TBME (1.35 mL) were placed in a 10 mL glass tube containing a magnetic stirring bar. A solution of Ph-SMAP (1.3 mg, 0.006 mmol, 2.0 mol%) in TBME (0.65 mL), a solution of [Ir(OMe)(cod)]₂ (2.0 mg, 0.0030 mmol, 1.0 mol%) in TBME (1.0 mL) and 2-ethylpyridine (**1a**) (96.4 mg, 0.90 mmol) were added successively. The tube was sealed with a screw cap and removed from the glovebox. The reaction mixture was stirred at 25 °C for 12 h. Solvent was removed under reduced pressure. An internal standard (1,1,2,2-tetrachloroethane) was added to the residue. The yield of the product **3a** was determined by ¹H NMR (0%).

Typical Procedure for the Borylation of 2-Alkylpyridine Derivatives (Table 2, Entry 1). In a glove box, Silica-SMAP (0.070 mmol/g, 85.7 mg, 0.0060 mmol, 2.0 mol%), bis(pinacolato)diboron (**2**) (76.2 mg, 0.30 mmol), and anhydrous, degassed hexane (1.0 mL) were placed in a 10 mL glass tube containing a magnetic stirring bar. A solution of [Ir(OMe)(cod)]₂ (2.0 mg, 0.0030 mmol, 1.0 mol%) in hexane (2.0 mL) and 2-isopropylpyridine (**1b**) (109.0 mg, 0.9 mmol) were added successively. The tube was sealed with a screw cap and removed from the glove box. The reaction mixture was stirred at 60 °C for 12 h, and filtered through a glass pipet equipped with a cotton filter. Solvent was removed under reduced pressure. An internal standard (1,1,2,2-tetrachloroethane) was

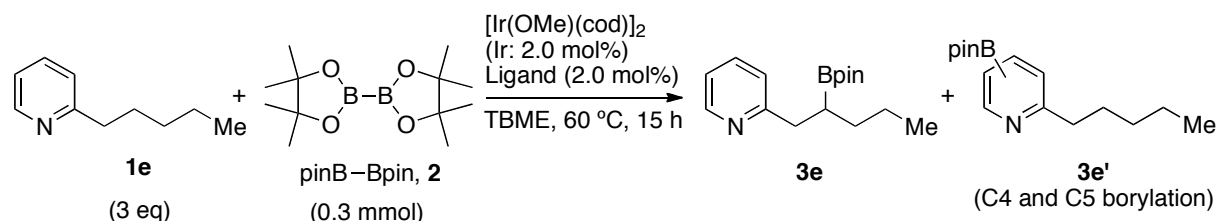
added to the residue. The yield of the product **3b** was determined by ^1H NMR (109%). The crude material was then purified by Kugelrohr distillation, to give the corresponding product **3b** (52.0 mg, 0.22 mmol in 73% isolated yield).

Procedure for the One-Carbon Homologation Following Oxidation of 3f (Scheme 2, Upper Side). Under Ar atmosphere, **3f** (64.7 mg, 0.20 mmol), bromochloromethane (51.7 mg, 0.4 mmol), and anhydrous THF (2.0 mL) were placed in a 10 mL glass tube containing a magnetic stirring bar. The tube was sealed with a screw cap with a Teflon-coated silicon rubber septum. After the mixture was cooled to -78°C , $n\text{-BuLi}$ in hexane (1.6 M, 24.4 μL , 0.4 mmol) was added. The mixture was stirred at -78°C for 5 min, and stirred at room temperature for 30 min. After the reaction, solvent was removed under reduced pressure. An internal standard (1,1,2,2-tetrachloroethane) was added to the residue and the yield of desired product 2-[2-benzyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propyl]-pyridine (**5**) was determined by ^1H NMR (56%) in the crude mixture. The resulting product **5** was used to next reaction without further purification. The boronate **5**, THF (0.6 mL), water (0.6 mL) and sodium perborate tetrahydrate (51.7 mg, 0.34 mmol) were placed in a round bottom flask, and the reaction mixture was stirred vigorously at room temperature for 2 h under air. After the reaction, solvent and water were evaporated under reduced pressure. The concentrate was suspended in CHCl_3 , filtered through a pipet equipped with cotton. The filtrate was concentrated under reduced pressure, and the crude mixture was then purified by silica gel chromatography to give the corresponding alcohol **6** (24.5 mg, 0.11 mmol in 54% isolated yield in two steps).

Procedure for the Oxidation of 3h (Scheme 2, Bottom Side). The oxidation of **3h** was performed according to the reported procedure.⁴ THF (0.5 mL), water (0.5 mL), and sodium perborate tetrahydrate (46.1 mg, 0.3 mmol) were successively added to a vial tube containing **3h**. The reaction mixture was stirred vigorously at room temperature for 2 h under air. After the solvent and water were evaporated under reduced pressure, the mixture was suspended in CHCl_3 , and the suspension was filtered through a pipet equipped with cotton. The filtrate was concentrated under reduced pressure and the crude mixture was then purified by silica gel chromatography to give the corresponding alcohol **7** (13.1 mg, 0.74 mmol in 74% isolated yield). The trans stereo-configuration was determined by comparing the ^1H NMR spectrum of **3h** with that of the reported compound.⁵

Ligand Effect

Table S1. Ligand Screening for Ir-Catalyzed Borylation of 2-Pentylpyridine (**1e**).^a



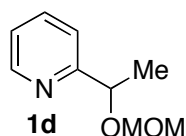
entry	ligand	NMR yield (%) ^b	
		3e	3e'
1	Silica-SMAP	106 (79)	11
2	Silica-TRIP	89	11
3	Ph-SMAP	0	0
4	PMe ₃	0	0
5	PCy ₃	6	5
6	P ^t Bu ₃	0	0
7	PPh ₃	0	0
8	XPhos	0	0
9	dtbpy	0	154 ^c
10	none	0	0

^a Conditions: **1e** (0.9 mmol), **2** (0.3 mmol), $[\text{Ir}(\text{OMe})(\text{cod})]_2$ (Ir: 0.0060 mmol), ligand (0.0060 mmol), TBME (3.0 mL), 60 °C, 15 h. ^b ¹H-NMR yield based on **2**. Isolated yield is shown in parentheses. ^c C4 borylation 97% and C5 borylation 47%.

Compounds Characterization

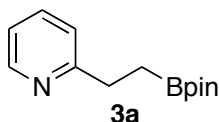
The substrates for borylation **1a**, **1e**, **1f** are known compounds. The substrates **1b**⁶, **1c**⁶, **1g**⁷ and **1h**⁸, and alcohol **7**⁵ are reported in the literatures.

Preparation of 2-[1-(Methoxymethoxy)ethyl]pyridine (1d). 2-[1-(Methoxymethoxy)ethyl]-pyridine (**1d**) was prepared via the protection of 2-[1-(hydroxy)ethyl]pyridine with methoxymethyl group.



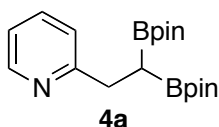
Colorless oil. ¹H NMR (CDCl₃) δ 1.53 (d, *J* = 6.6 Hz, 3H), 3.38 (s, 3H), 4.63 (d, *J* = 6.6 Hz, 1H), 4.72 (d, *J* = 6.6 Hz, 1H), 4.86 (q, *J* = 6.6 Hz, 1H), 7.19 (ddd, *J* = 7.8, 5.7, 0.9 Hz, 1H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.70 (td, *J* = 7.8, 1.8 Hz, 1H), 8.57 (dd, *J* = 5.7, 0.9 Hz, 1H). ¹³C NMR (CDCl₃) δ 22.00, 55.43, 75.46, 94.91, 120.27, 122.37, 136.75, 149.24, 162.93. HRMS-ESI (*m/z*): [M+Na]⁺ Calcd for C₉H₁₃O₂NNa, 190.08385; found, 190.08394.

2-[2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl]pyridine (3a)



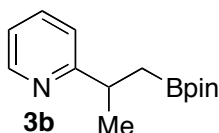
Colorless oil. $^1\text{H NMR}$ (CDCl_3) δ 1.08 (t, $J = 7.8$ Hz, 2H), 1.25 (s, 12H), 2.99 (t, $J = 7.8$ Hz, 2H), 7.18 (t, $J = 6.3$ Hz, 1H), 7.25 (d, $J = 7.8$ Hz, 1H), 7.67 (td, $J = 7.8, 1.5$ Hz, 1H), 8.54 (d, $J = 4.5$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 25.12 (4C), 31.74, 81.69 (2C), 121.42, 122.74, 137.75, 146.35, 163.73. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–APCI** (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{21}\text{N}^{10}\text{BO}_2$, 233.16964; found, 233.16962.

2-[2,2-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl]pyridine (4a)



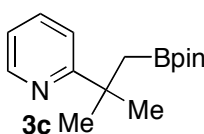
White solid. **M.p.** 75.6–77.3 °C. $^1\text{H NMR}$ (CDCl_3) δ 0.93 (t, $J = 8.1$ Hz, 1H), 1.24 (s, 12H), 1.25 (s, 12H), 3.19 (d, $J = 8.1$ Hz, 2H), 7.25 (m, 1H), 7.32 (d, $J = 7.8$ Hz, 1H), 7.74 (td, $J = 7.8, 1.8$ Hz, 1H), 8.56 (d, $J = 5.4$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 25.30 (4C), 25.58 (4C), 33.58, 81.53 (4C), 121.83, 123.06, 139.07, 143.59, 164.00. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{19}\text{H}_{32}\text{N}^{10}\text{B}_2\text{O}_4$, 358.25846; found, 358.25870.

2-[1-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-propan-2-yl]pyridine (3b)



Colorless oil. $^1\text{H NMR}$ (CDCl_3) δ 0.79 (dd, $J = 14.1, 7.5$ Hz, 1H), 1.14 (dd, $J = 14.1, 7.5$ Hz, 1H), 1.22 (s, 6H), 1.26 (s, 6H), 1.36 (d, $J = 7.5$ Hz, 3H), 3.28 (sext, $J = 7.5$ Hz, 1H), 7.27 (t, $J = 6.3$ Hz, 1H), 7.38 (d, $J = 7.5$ Hz, 1H), 7.79 (td, $J = 7.5, 1.5$ Hz, 1H), 8.57 (d, $J = 5.1$ Hz, 1H). $^{13}\text{C NMR}$ (CDCl_3) δ 21.78, 25.39 (2C), 25.58 (2C), 37.02, 80.54 (2C), 121.52, 121.87, 139.19, 144.02, 167.68. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{14}\text{H}_{23}\text{N}^{10}\text{BO}_2$, 247.18527; found, 247.18544.

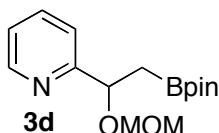
2-[2-Methyl-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-2-yl]pyridine (3c)



White solid. **M.p.** 72.5–74.0 °C. $^1\text{H NMR}$ (CDCl_3) δ 0.88 (s, 2H), 1.26 (s, 12H), 1.38 (s, 6H), 7.36 (ddd, $J = 8.1, 5.7, 1.2$ Hz, 1H), 7.47 (d, $J = 8.1$ Hz, 1H), 7.89 (td, $J = 7.8, 1.2$ Hz, 1H), 8.58 (d,

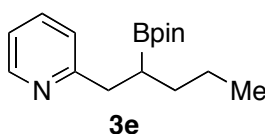
$J = 5.7$ Hz, 1H). ^{13}C NMR (CDCl_3) δ 25.93 (4C), 31.07 (2C), 41.40, 79.32 (2C), 120.82, 122.56, 140.98, 141.59, 171.34. A signal for the carbon directly attached to the boron atom was not observed. HRMS–ESI (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{15}\text{H}_{25}^{10}\text{BNO}_2$, 261.20092; found, 261.20113.

2-[1-(Methoxymethoxy)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl]pyridine (3d)



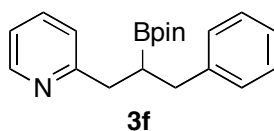
Colorless oil. ^1H NMR (CDCl_3) δ 1.16 (dd, $J = 13.5, 9.0$ Hz, 1H), 1.21 (s, 6H), 1.25 (s, 6H), 1.53 (dd, $J = 13.5, 6.9$ Hz, 1H), 3.41 (s, 3H), 4.71 (d, $J = 6.6$ Hz, 1H), 4.86 (dd, $J = 6.6$ Hz, 1H), 5.12 (dd, $J = 9.0, 6.9$ Hz, 1H), 7.29 (t, $J = 6.3$ Hz, 1H), 7.59 (d, $J = 7.8$ Hz, 1H), 7.80 (td, $J = 7.8, 1.5$ Hz, 1H), 8.57 (d, $J = 5.1$ Hz, 1H). ^{13}C NMR (CDCl_3) δ 25.01 (2C), 25.22 (2C), 55.50, 77.24, 81.53 (2C), 94.97, 121.44, 122.89, 138.50, 145.41, 162.79. A signal for the carbon directly attached to the boron atom was not observed. HRMS–ESI (m/z): $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{24}\text{O}_4\text{N}^{10}\text{BNa}$, 315.17269; found, 315.17269. Isolated product is contaminated with a trace of **1d**.

2-[2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)pentyl]pyridine (3e)



Colorless oil. ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.1$ Hz, 3H), 1.12–1.18 (m, 2H), 1.23 (s, 6H), 1.24 (s, 6H), 1.29–1.40 (m, 2H), 1.42–1.55 (m, 1H), 2.82 (dd, $J = 16.2, 6.6$ Hz, 1H), 3.07 (dd, $J = 16.2, 6.6$ Hz, 1H), 7.22 (t, $J = 5.7$ Hz, 1H), 7.28 (d, $J = 7.4$ Hz, 1H), 7.71 (td, $J = 7.4, 0.9$ Hz, 1H), 8.55 (d, $J = 7.1$ Hz, 1H). ^{13}C NMR (CDCl_3) δ 14.26, 22.24, 25.49 (2C), 25.60 (2C), 33.27, 38.09, 80.87 (2C), 121.66, 123.48, 138.44, 144.86, 162.72. A signal for the carbon directly attached to the boron atom was not observed. HRMS–ESI (m/z): $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{27}\text{O}_2\text{N}^{10}\text{B}$, 275.21657; found, 275.21659.

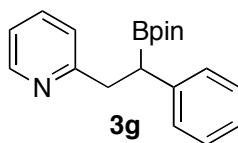
2-[3-Phenyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propyl]pyridine (3f)



Colorless oil. ^1H NMR (CDCl_3) δ 1.28 (s, 12H), 1.48–1.56 (m, 1H), 2.29 (t, $J = 14.4$ Hz, 1H), 2.74 (dd, $J = 17.1, 5.7$ Hz, 1H), 2.97 (dd, $J = 17.1, 7.1$ Hz, 1H), 3.06 (dd, $J = 14.4, 4.5$ Hz, 1H), 7.15–7.29 (m, 6H), 7.34 (t, $J = 6.3$ Hz, 1H), 7.80 (td, $J = 7.8, 1.5$ Hz, 1H), 8.61 (d, $J = 5.4$ Hz, 1H). ^{13}C NMR (CDCl_3) δ 25.90 (2C), 26.13 (2C), 36.58, 36.75, 80.20 (2C), 122.29, 123.75, 125.27, 128.14 (2C), 128.85 (2C), 139.84, 143.07, 143.94, 162.55. A signal for the carbon directly attached

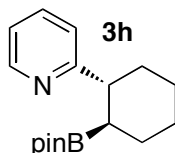
to the boron atom was not observed. **HRMS–ESI** (m/z): $[M+H]^+$ Calcd for $C_{20}H_{27}N^{10}BO_2$, 323.21657; found, 323.21662.

2-[2-Phenyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl]pyridine (3g)



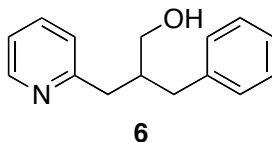
White solid. **M.p.** 114.2–116.1°C. **1H NMR** ($CDCl_3$) δ 1.02 (s, 6H), 1.09 (s, 6H), 2.51 (t, J = 8.4 Hz, 1H), 3.40 (d, J = 8.4 Hz, 2H), 7.09 (m, 1H), 7.21–7.23 (m, 4H), 7.38–7.46 (m, 2H), 7.89 (td, J = 7.8, 1.5 Hz, 1H), 8.60 (d, J = 5.4 Hz, 1H). **^{13}C NMR** ($CDCl_3$) δ 25.74 (2C), 26.37 (2C), 39.05, 80.13 (2C), 122.75, 123.44, 124.16, 127.69 (2C), 128.14 (2C), 140.42, 142.41, 145.83, 162.17. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[M+H]^+$ Calcd for $C_{19}H_{25}O_2N^{10}B$, 309.20092; found, 309.20093.

2-[2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)cyclohexyl]pyridine (3h)



Colorless oil. **1H NMR** ($CDCl_3$) δ 0.97 (td, J = 12.0, 2.7 Hz, 1H), 1.11 (s, 6H), 1.17 (s, 6H), 1.21–1.61 (m, 4H), 1.77–1.90 (m, 3H), 2.17 (dd, J = 12.0, 3.3 Hz, 1H), 2.82 (td, J = 12.0, 3.3 Hz, 1H), 7.17 (tm, J = 6.6 Hz, 1H), 7.25 (d, J = 7.8 Hz, 1H), 7.69 (td, J = 7.8, 1.5 Hz, 1H), 8.52 (d, J = 4.5 Hz, 1H). **^{13}C NMR** ($CDCl_3$) δ 24.99 (2C), 25.33 (2C), 26.46, 26.90, 27.42, 31.94, 46.43, 81.02 (2C), 120.89, 121.33, 137.81, 145.55, 165.67. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): $[M+H]^+$ Calcd for $C_{17}H_{27}N^{10}BO_2$, 287.21657; found, 287.21664. The stereochemistry of **3h** was assigned by the coupling constant between the methine protons at C1 and C2 positions.

2-Benzyl-3-(pyridin-2-yl)propan-1-ol (6)



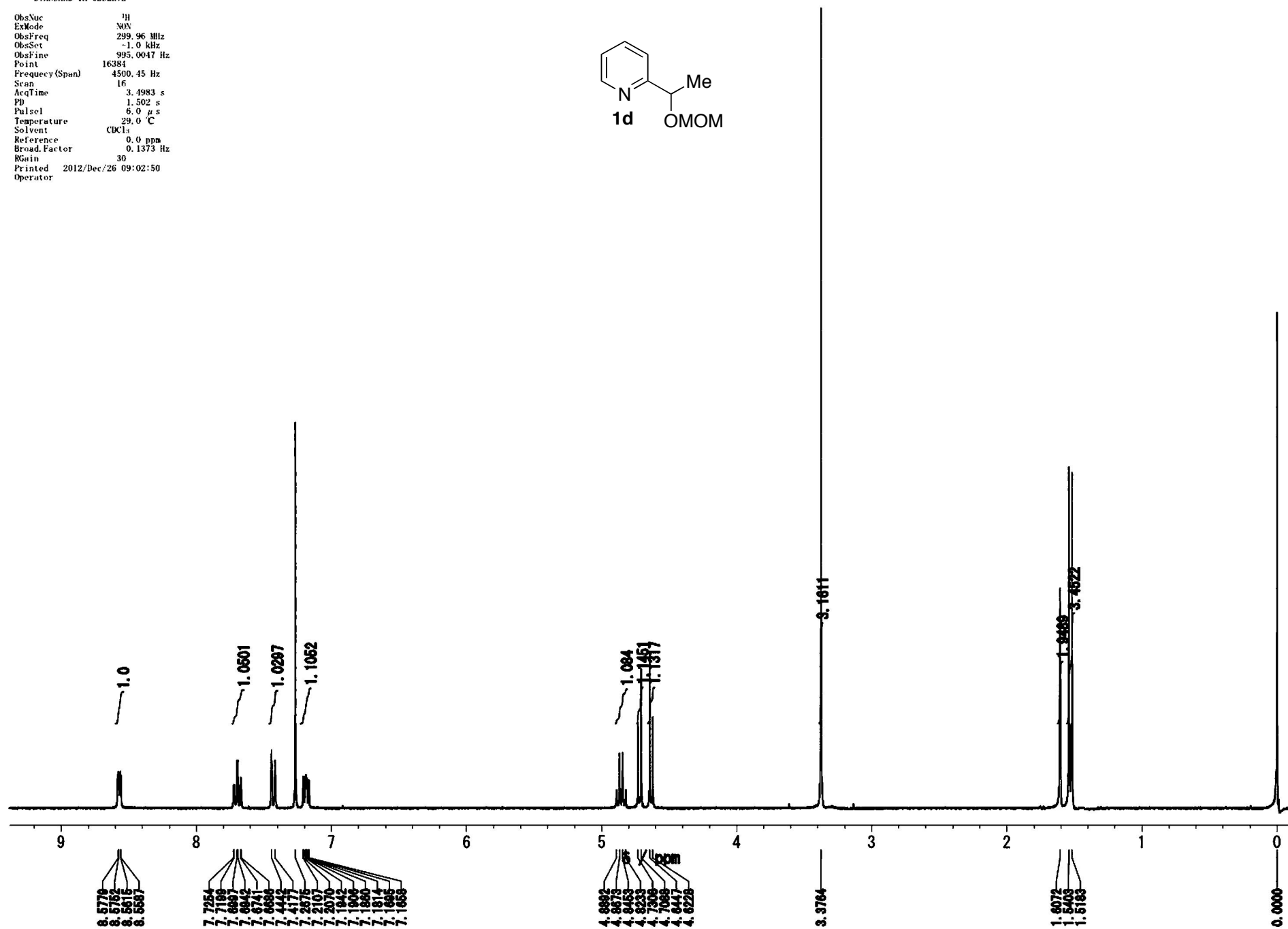
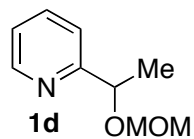
Colorless oil. **1H NMR** ($CDCl_3$) δ 2.34 (m, 1H), 2.59 (dd, J = 13.8, 7.5 Hz, 1H), 2.72 (dd, J = 13.8, 7.5 Hz, 1H), 2.93 (dd, J = 5.1, 3.0 Hz, 2H), 3.45 (dd, J = 11.1, 6.0 Hz, 1H), 3.59 (dd, J = 11.1, 6.0 Hz, 1H), 7.11 (d, J = 7.8, 1H), 7.15–7.23 (m, 5H), 7.29 (t, J = 7.5 Hz, 1H), 7.64 (td, J = 7.8, 1.5 Hz, 1H), 8.51 (d, J = 5.1 Hz, 1H). **^{13}C NMR** ($CDCl_3$) δ 38.78, 39.51, 42.68, 64.45, 121.45, 124.25, 126.08, 128.43 (2C), 129.20 (2C), 137.23, 140.45, 148.37, 160.16. **HRMS–ESI** (m/z): $[M+H]^+$ Calcd for $C_{15}H_{18}NO$, 228.138291; found, 228.13827.

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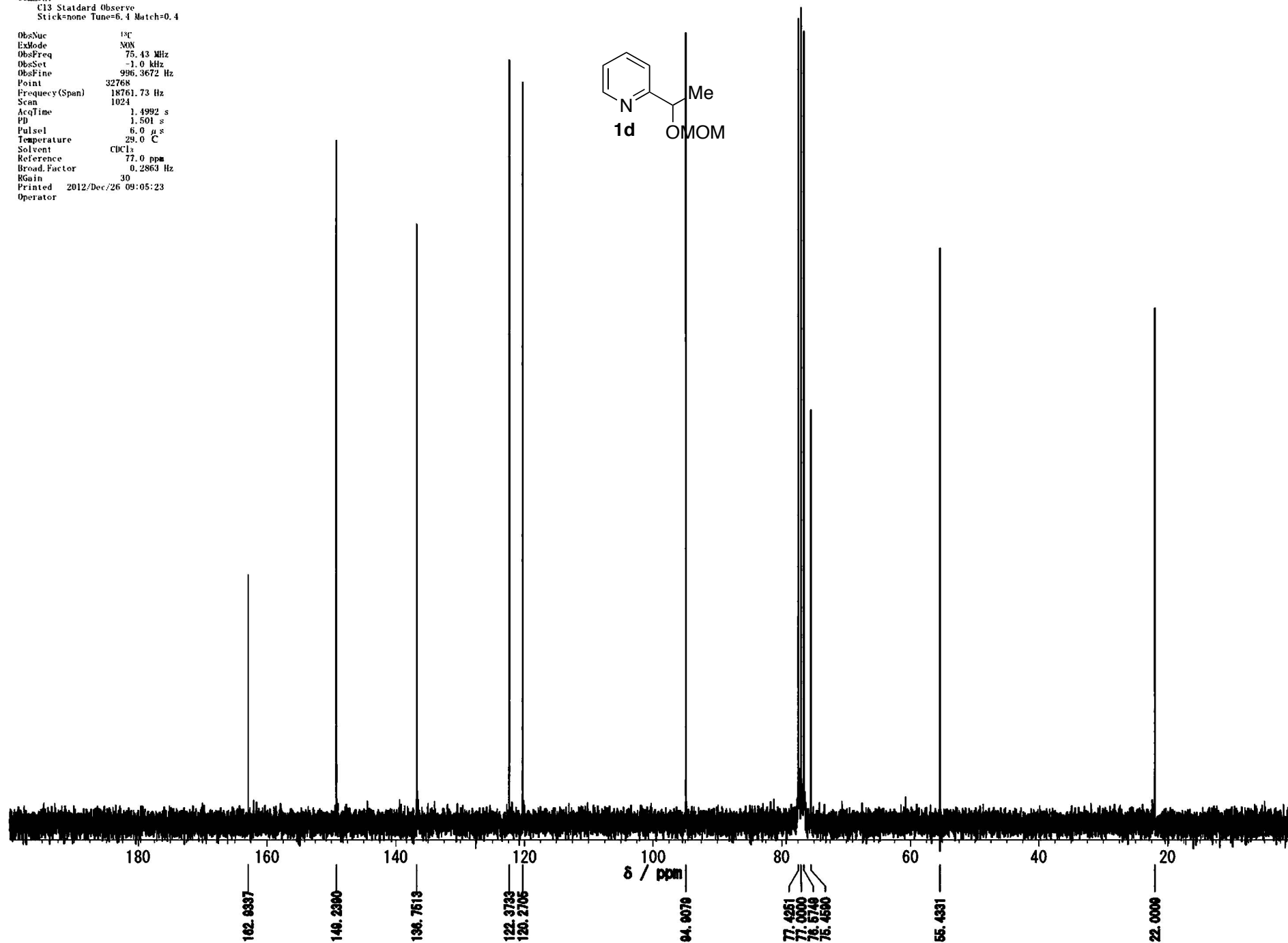
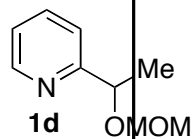
Date Oct 16 12
 Comment STANDARD 1H OBSERVE

ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency (Spun) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad. Factor 0.1373 Hz
 RGain 30
 Printed 2012/Dec/26 09:02:50
 Operator

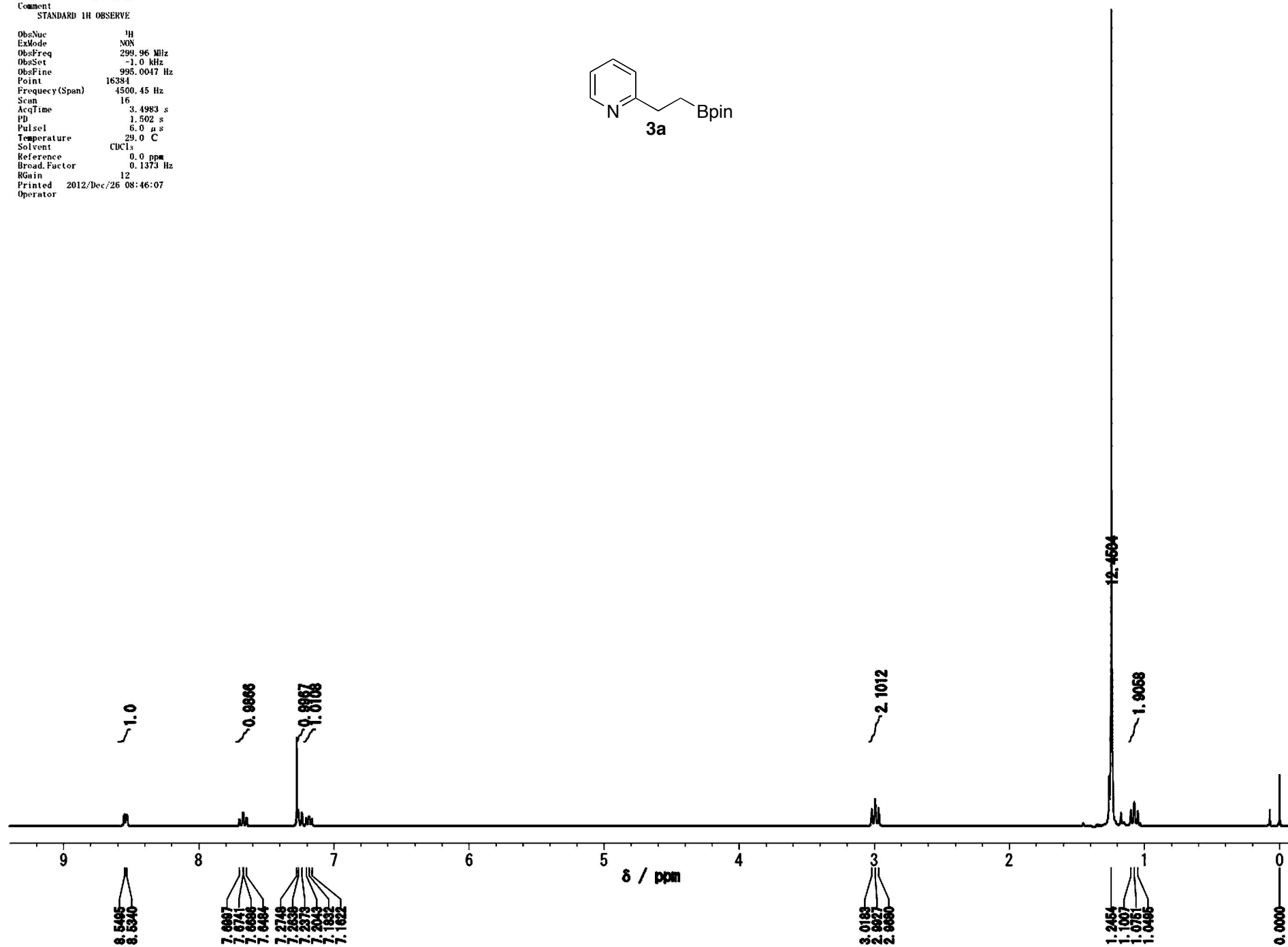
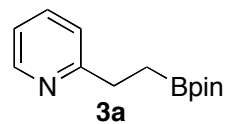


Date Dec 11 12
 Comment C13 Standard Observe
 Stick=None Tune=6.4 Match=0.4

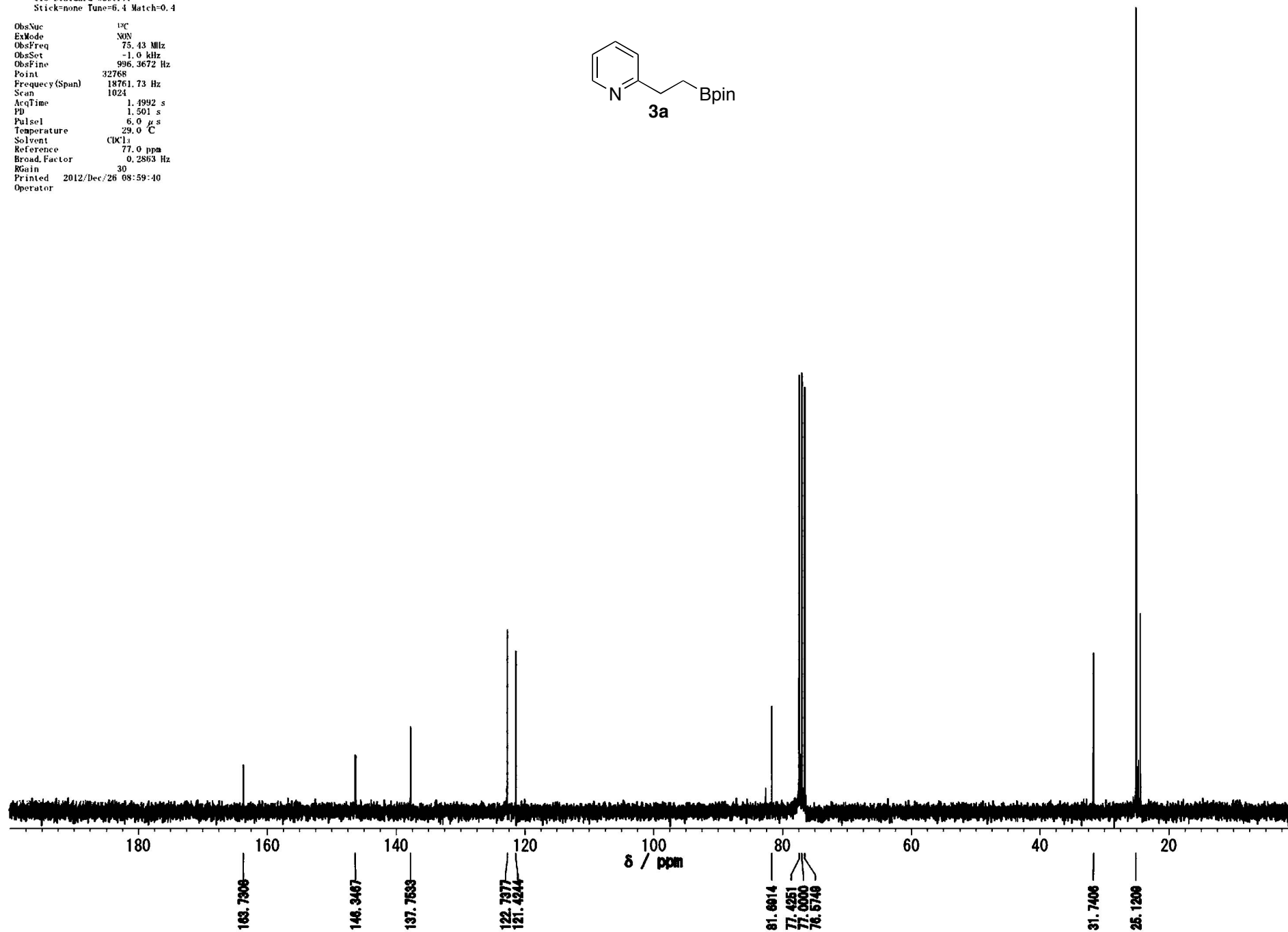
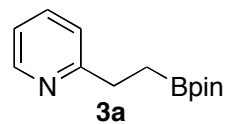
ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSv -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 09:05:23
 Operator



Date Oct 3 12
 Comment STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse1 6.0 μ s
 Temperature 29.0 C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad.Factor 0.1373 Hz
 RGain 12
 Printed 2012/Dec/26 08:46:07
 Operator

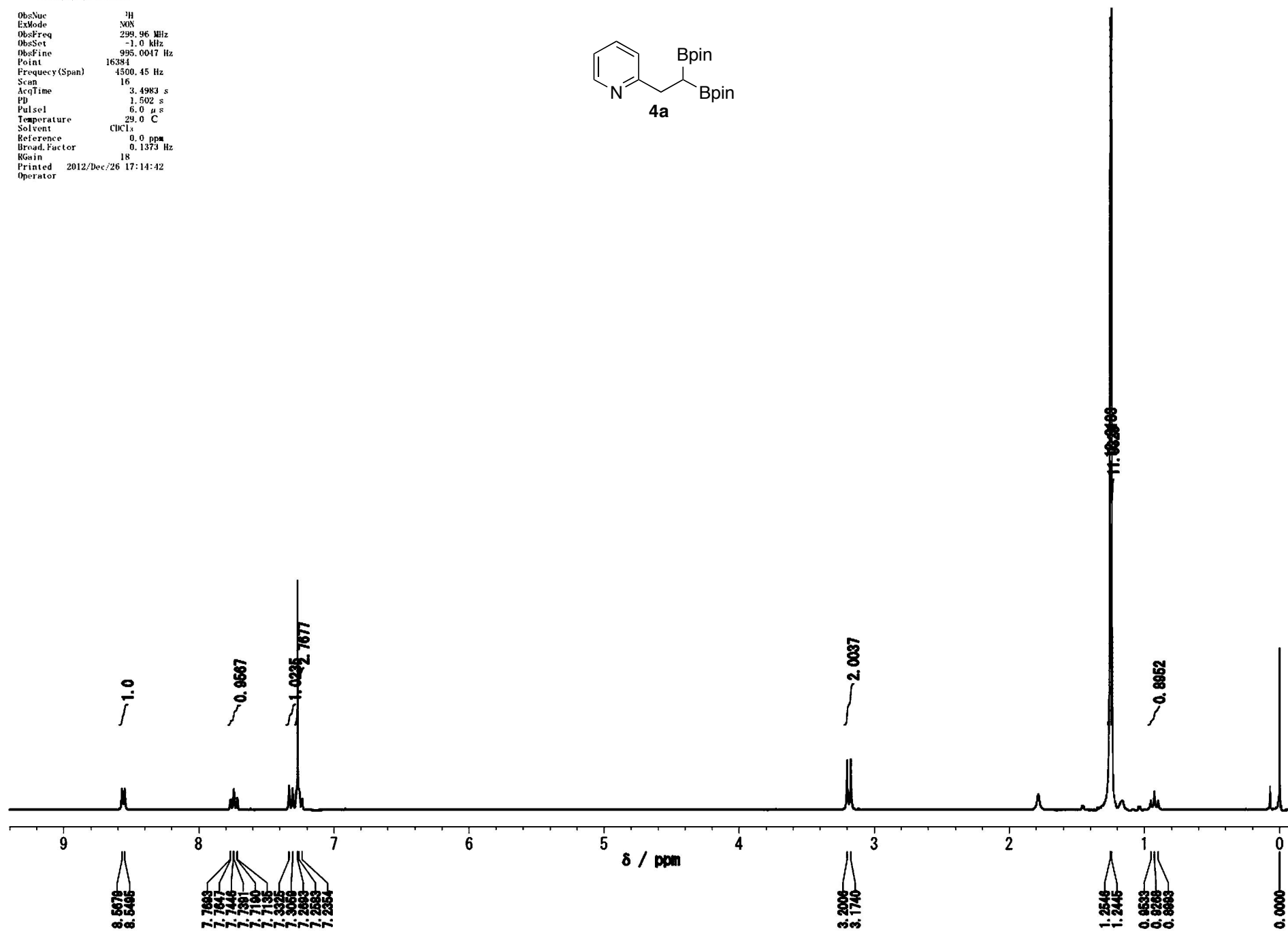
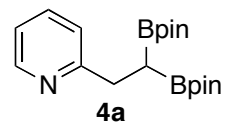


Date Oct 5 12
 Comment C13 Standard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 08:59:40
 Operator

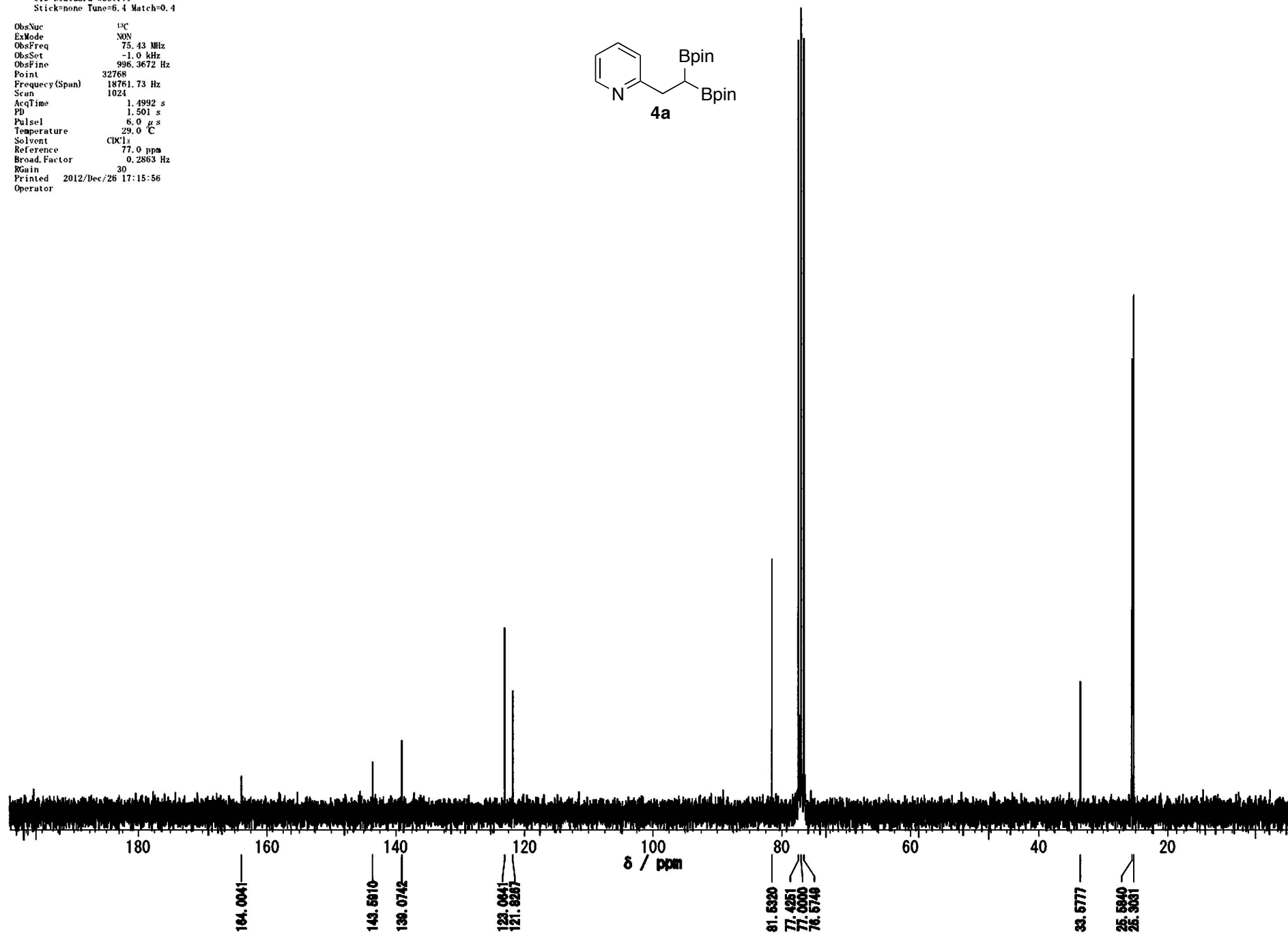
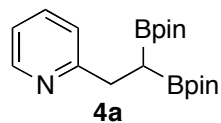


STANDARD 1H OBSERVE

ObsVac	3H
ExMode	NON
ObsFreq	299.96 MHz
ObsSet	-1.0 kHz
ObsFine	995.0047 Hz
Point	16384
Frequency(Span)	4300.45 Hz
Scan	16
AcqTime	3.4983 s
PD	1.502 s
Pulsel	6.0 μ s
Temperature	29.0 C
Solvent	CDCl ₃
Reference	0.0 ppm
Broad.Factor	0.1373 Hz
Kgain	1
Printed	2012/Dec/26 17:14:42
Operator	



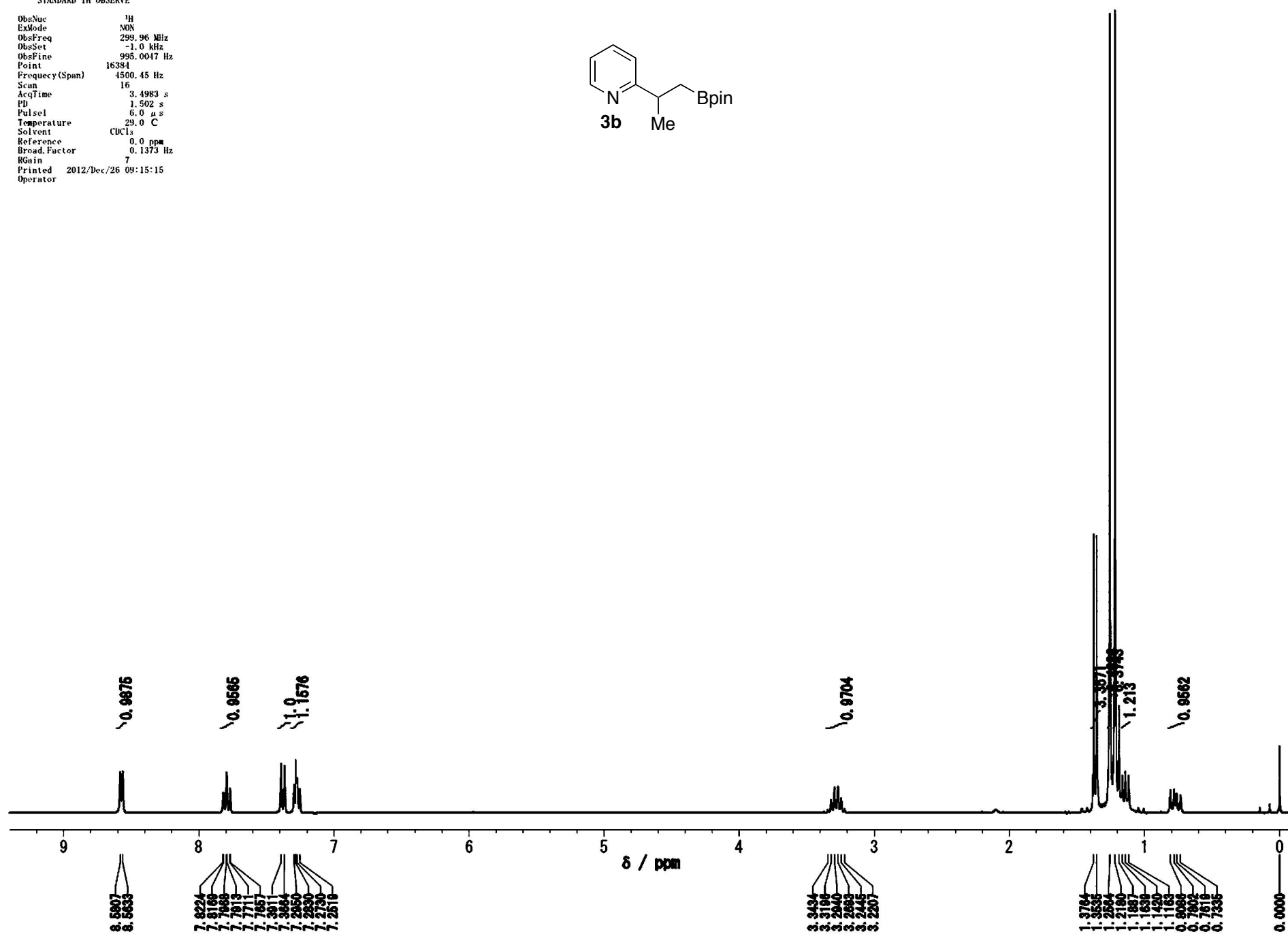
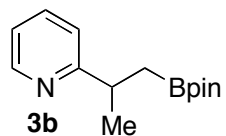
Date Oct 6 12
 Comment C13 Statdard Observe
 Stick:none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode XON
 ObsFreq 75.43 MHz
 ObsSrt -1.9 kHz
 ObsFme 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad. Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 17:15:56
 Operator



Date Oct 19 12

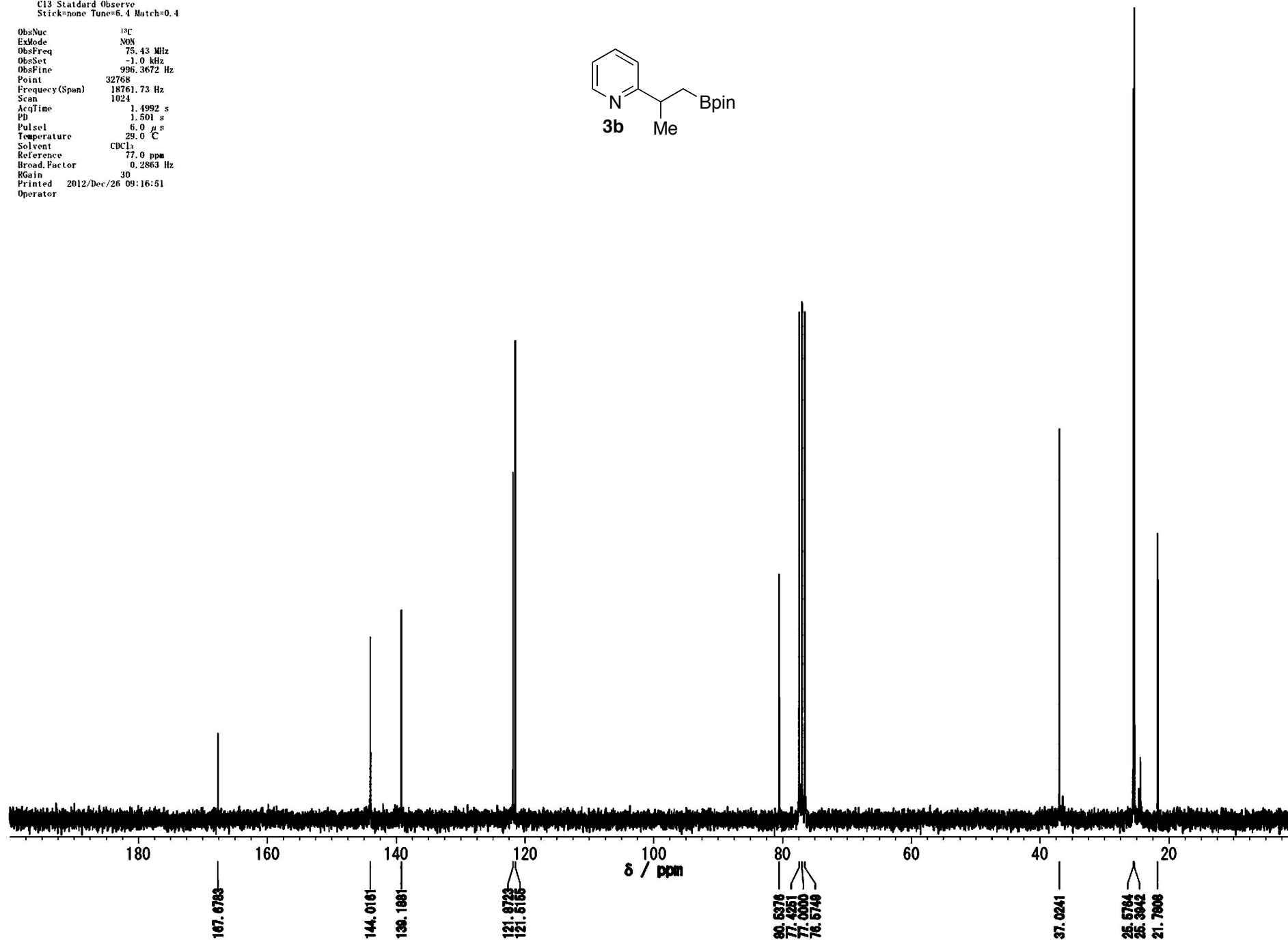
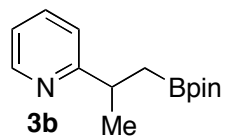
Comment STANDARD 1H OBSERVE

ObsNuc ¹H
ExMode MON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsFine 995.0047 Hz
Point 16384
Frequency(Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μ s
Temperature 29.0 C
Solvent CDCl₃
Reference 0.0 ppm
Broad.Factor 0.1373 Hz
RGain 7
Printed 2012/Dec/26 09:15:15
Operator



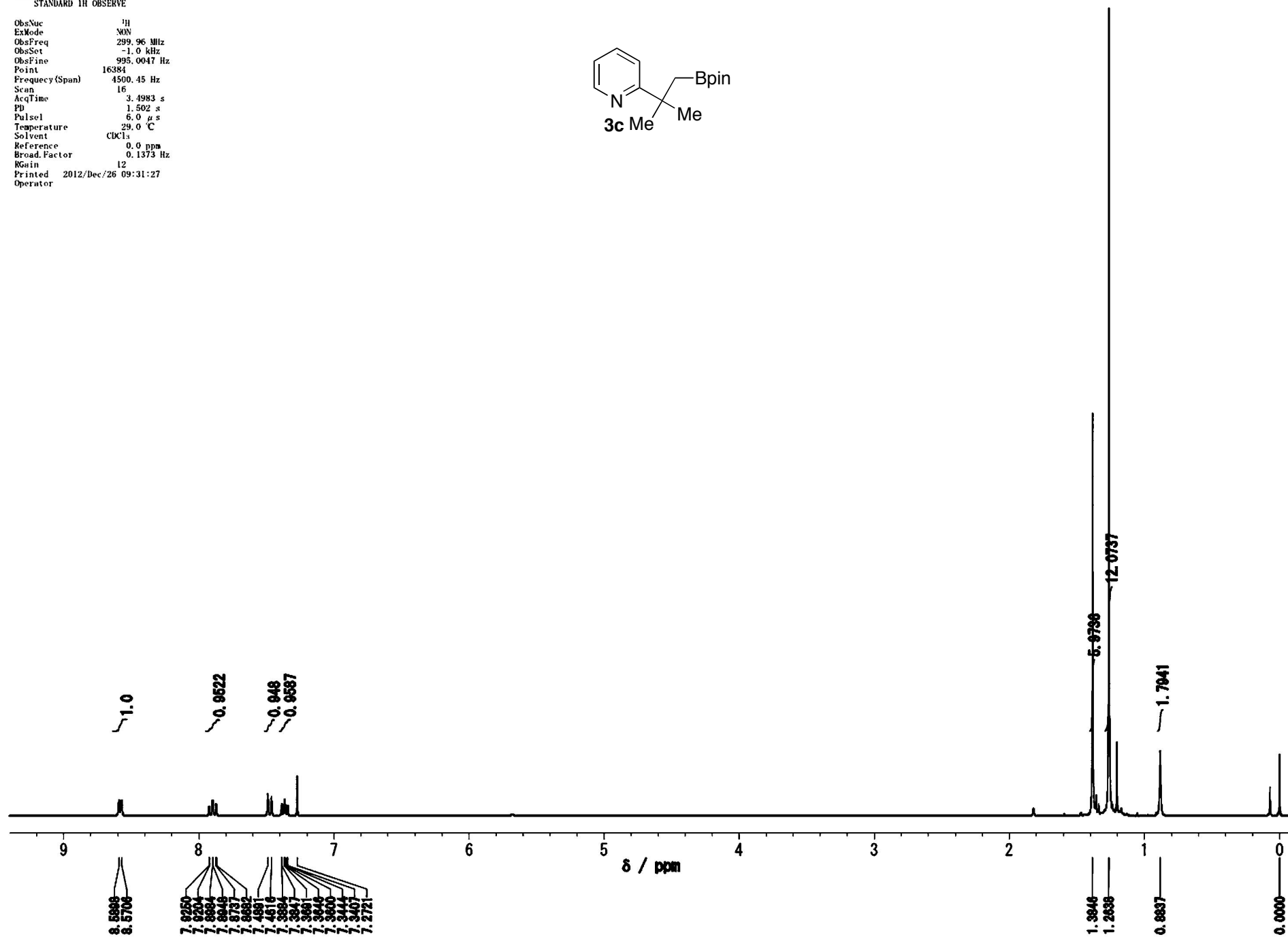
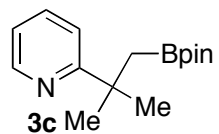
Date Oct 19 12
 Comment C13 Standard Observe
 Stick=None Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 09:16:51
 Operator

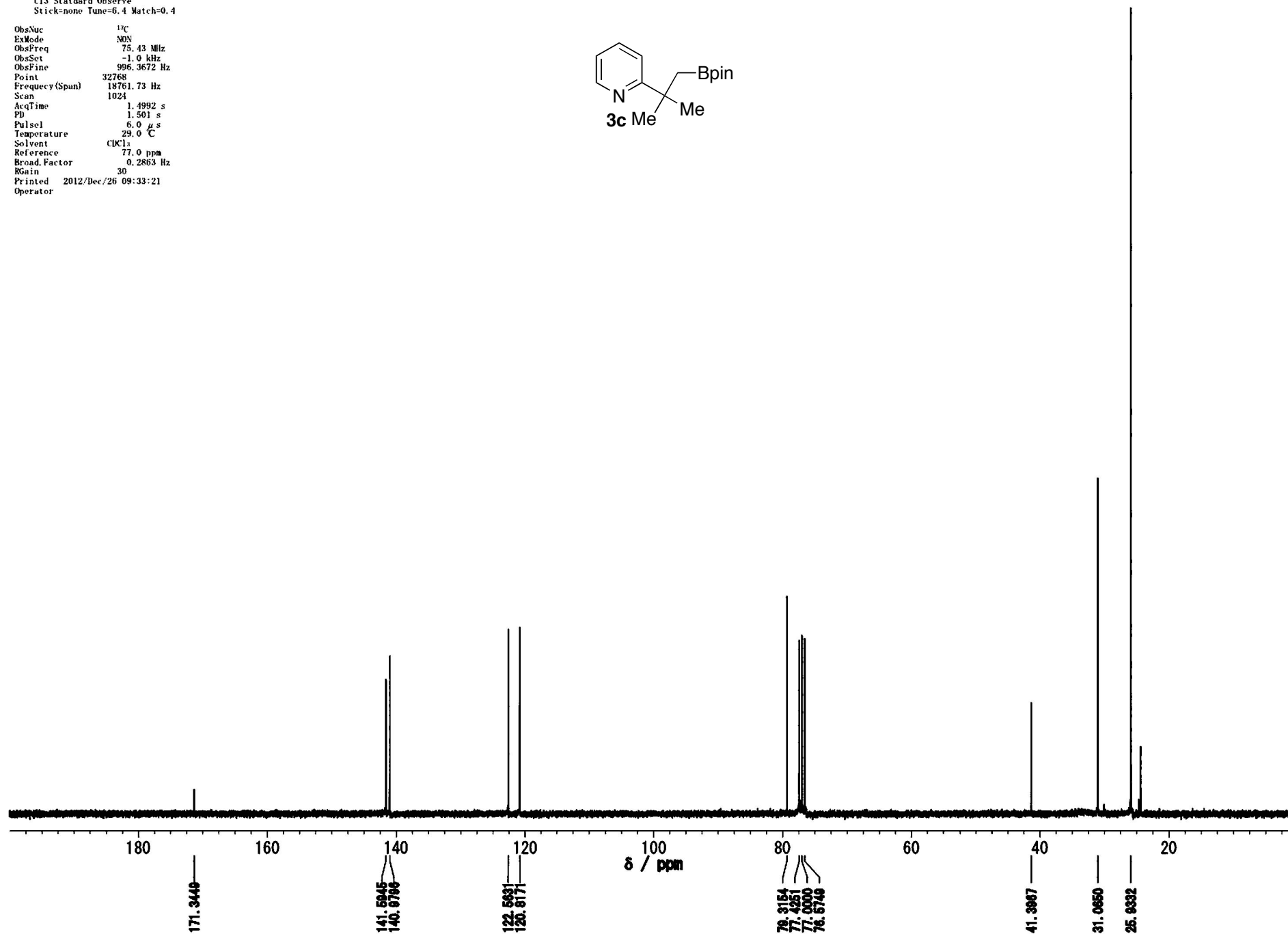
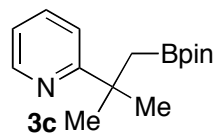


Date Oct 5 12
 Comment STANDARD 1H OBSERVE

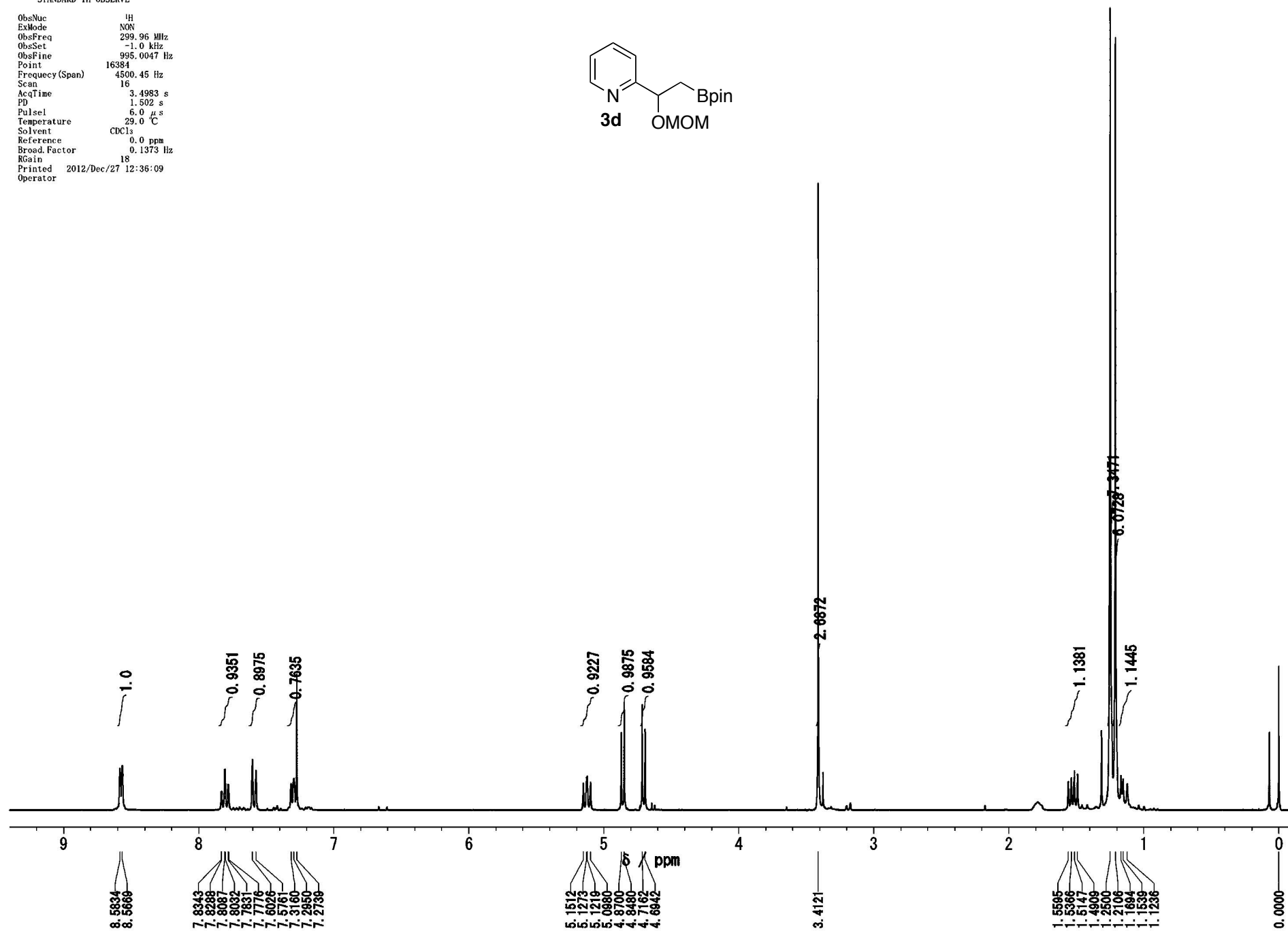
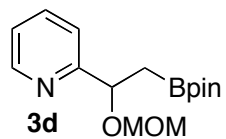
ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulsol 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 12
 Printed 2012/Dec/26 09:31:27
 Operator



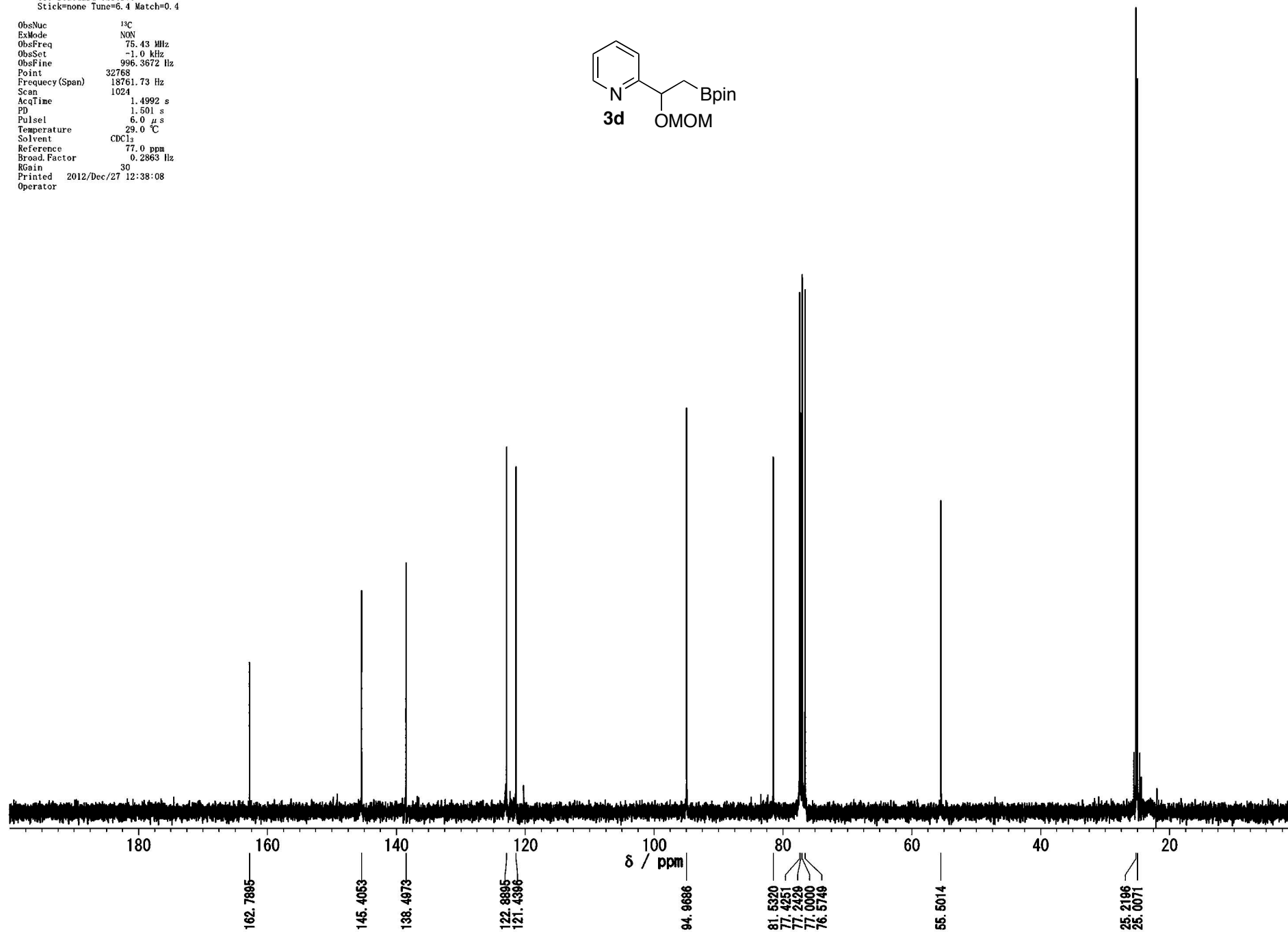
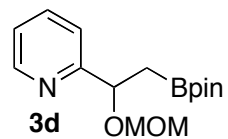
Date Nov 19 12
 Comment C13 Standard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ^{13}C
 ExMode NOX
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 995.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 09:33:21
 Operator



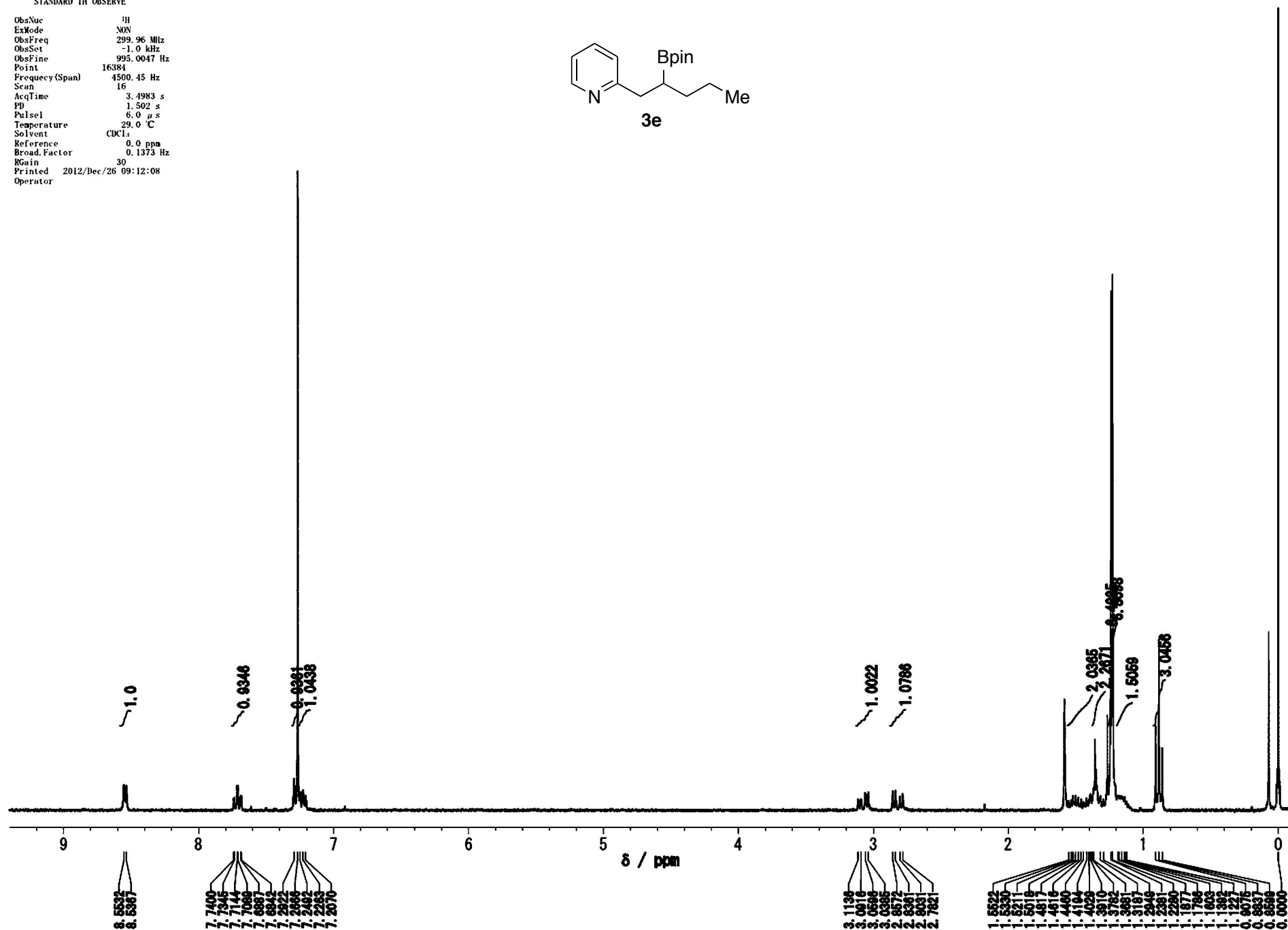
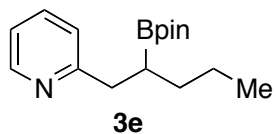
Date Nov 22 12
 Comment STANDARD 1H OBSERVE
 ObsNuc ¹H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad.Factor 0.1373 Hz
 RGain 18
 Printed 2012/Dec/27 12:36:09
 Operator



Date Dec 8 12
 Comment C13 Statdard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/27 12:38:08
 Operator

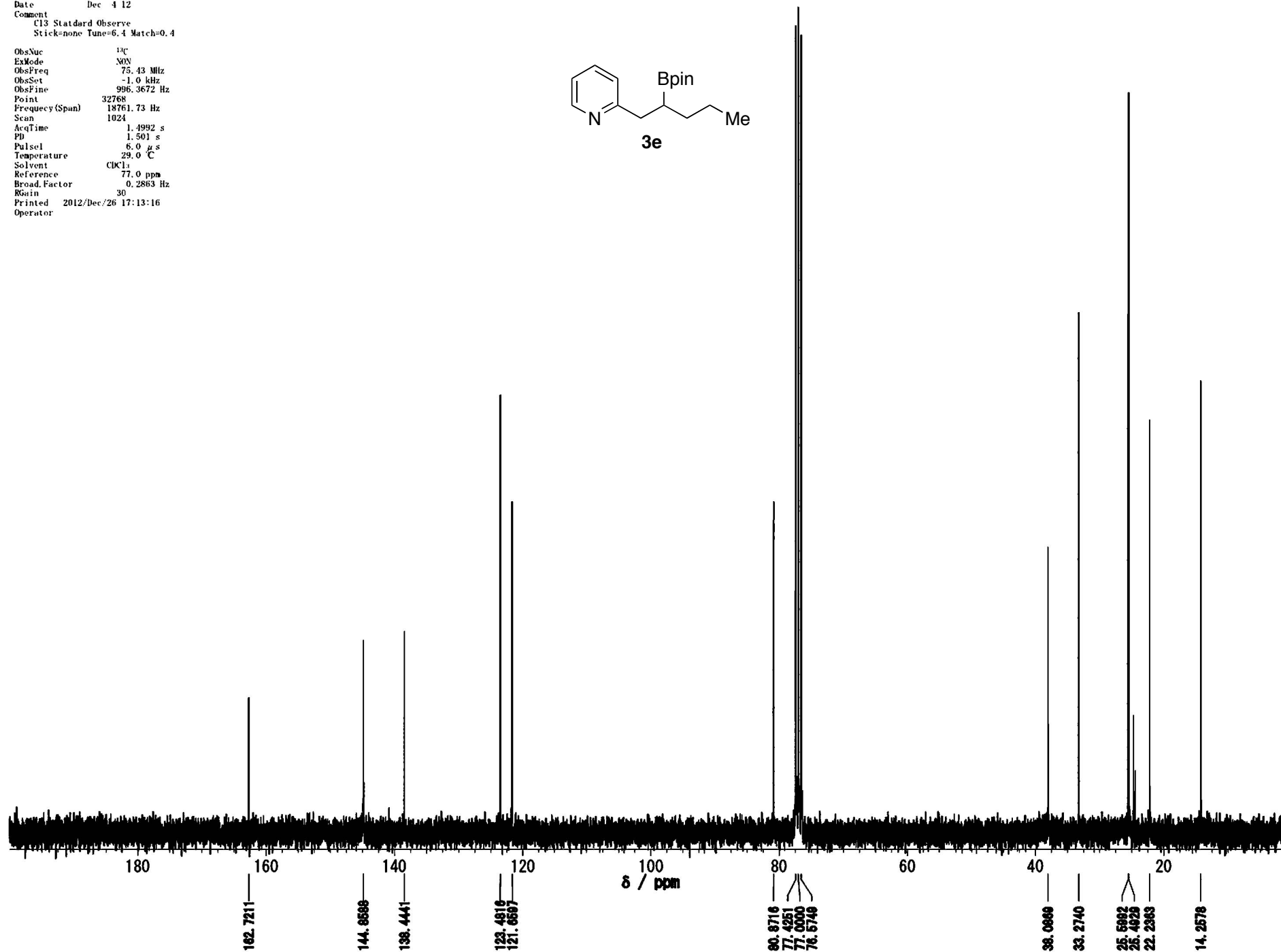
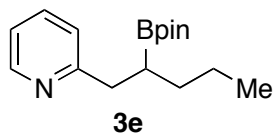


Date Dec 1 12
 Comment STANDARD 1H OBSERVE
 ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad.Factor 0.1373 Hz
 RGain 30
 Printed 2012/Dec/26 09:12:08
 Operator

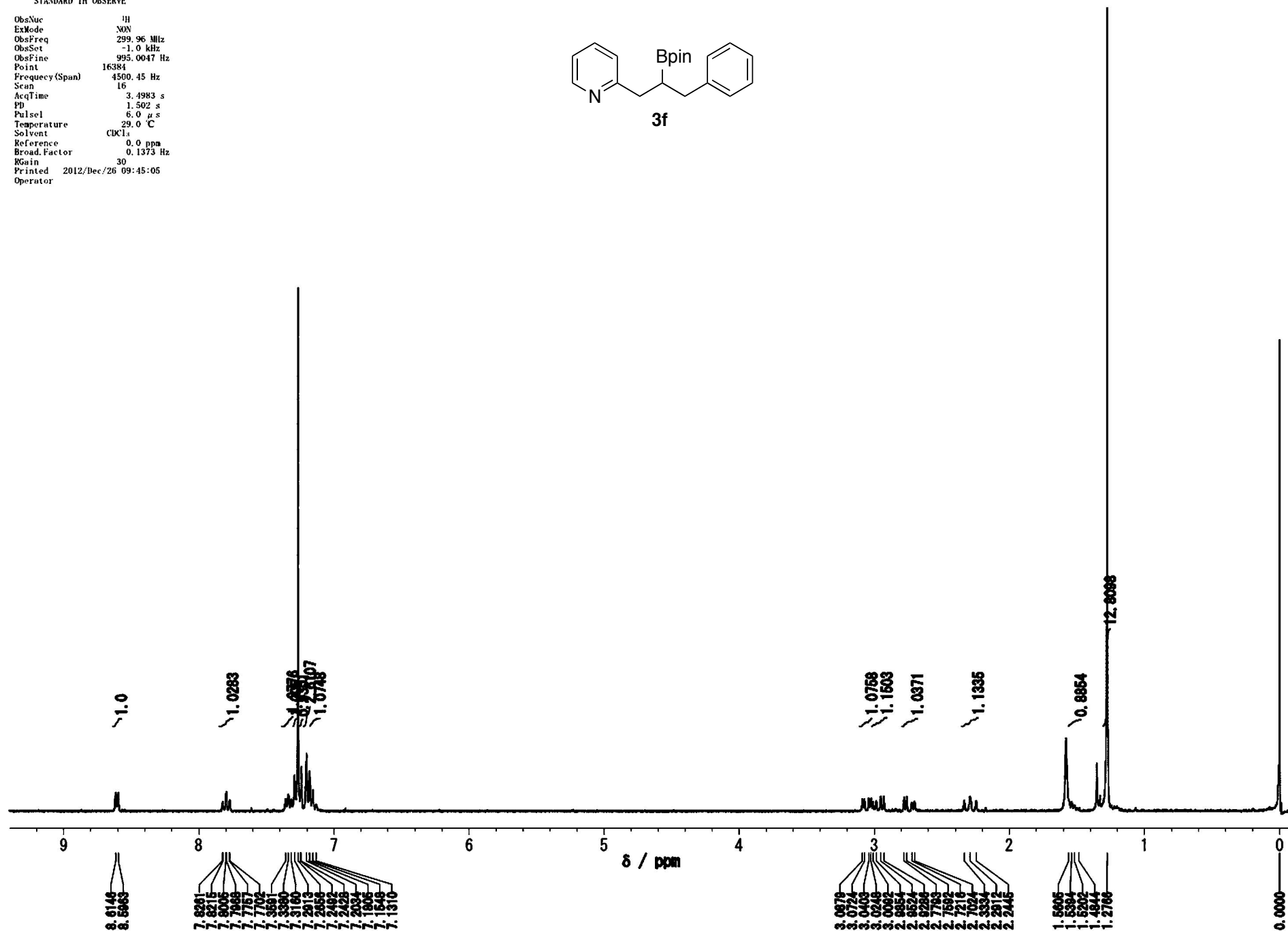
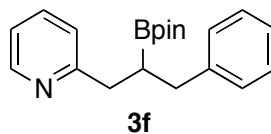


Date Dec 4 12
 Comment C13 Standard Observe
 Stick:none Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode XON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 995.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 17:13:16
 Operator

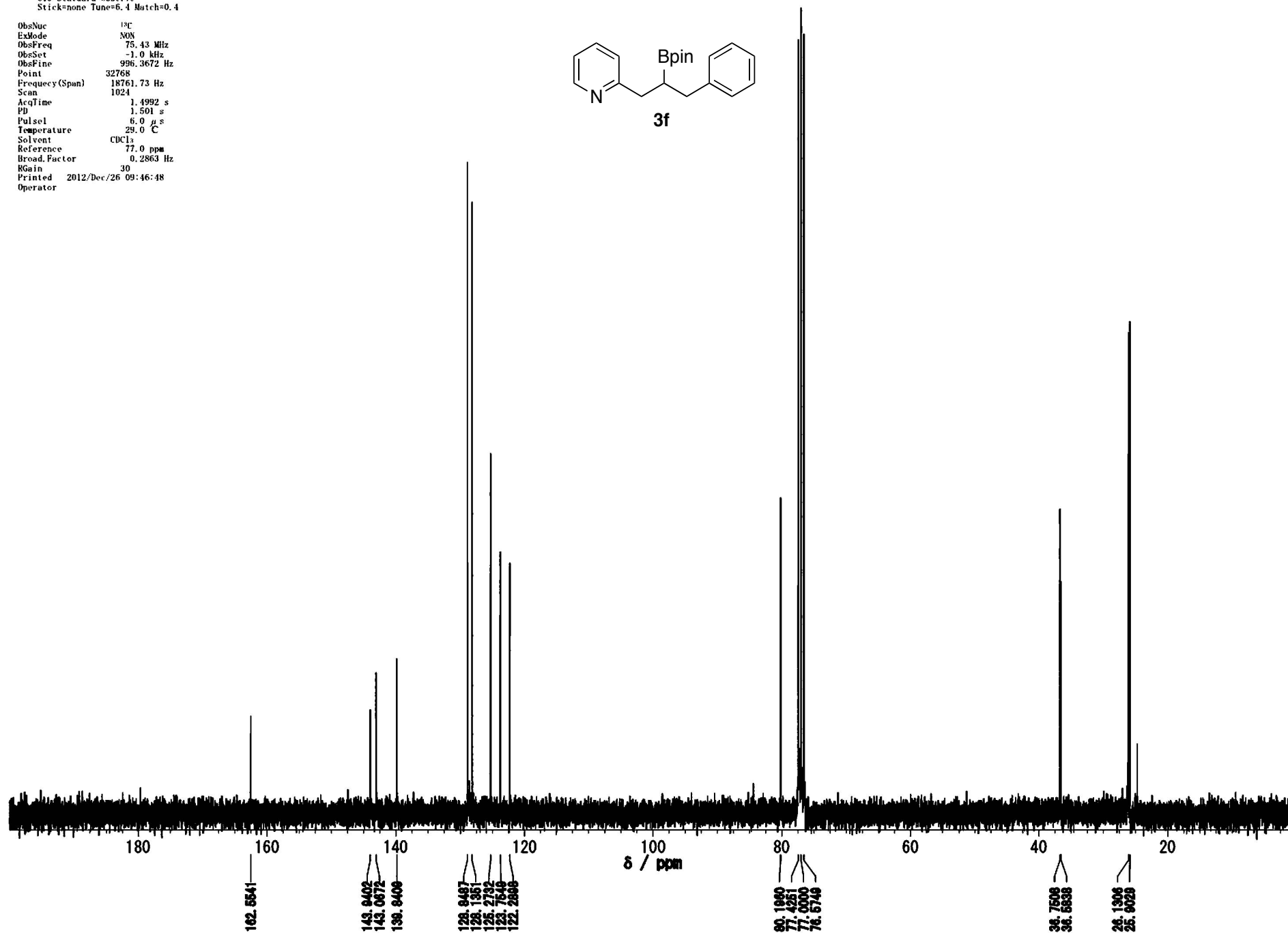
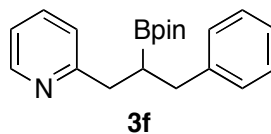


Date Dec 4 12
 Comment STANDARD 1H OBSERVE
 ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency(Span) 4500.45 Hz
 Scun 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulsel 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad.Factor 0.1373 Hz
 RGain 30
 Printed 2012/Dec/26 09:45:05
 Operator



Date Dec 5 12
 Comment C13 Standard Observe
 Stick=None Tune=6.4 Match=0.4

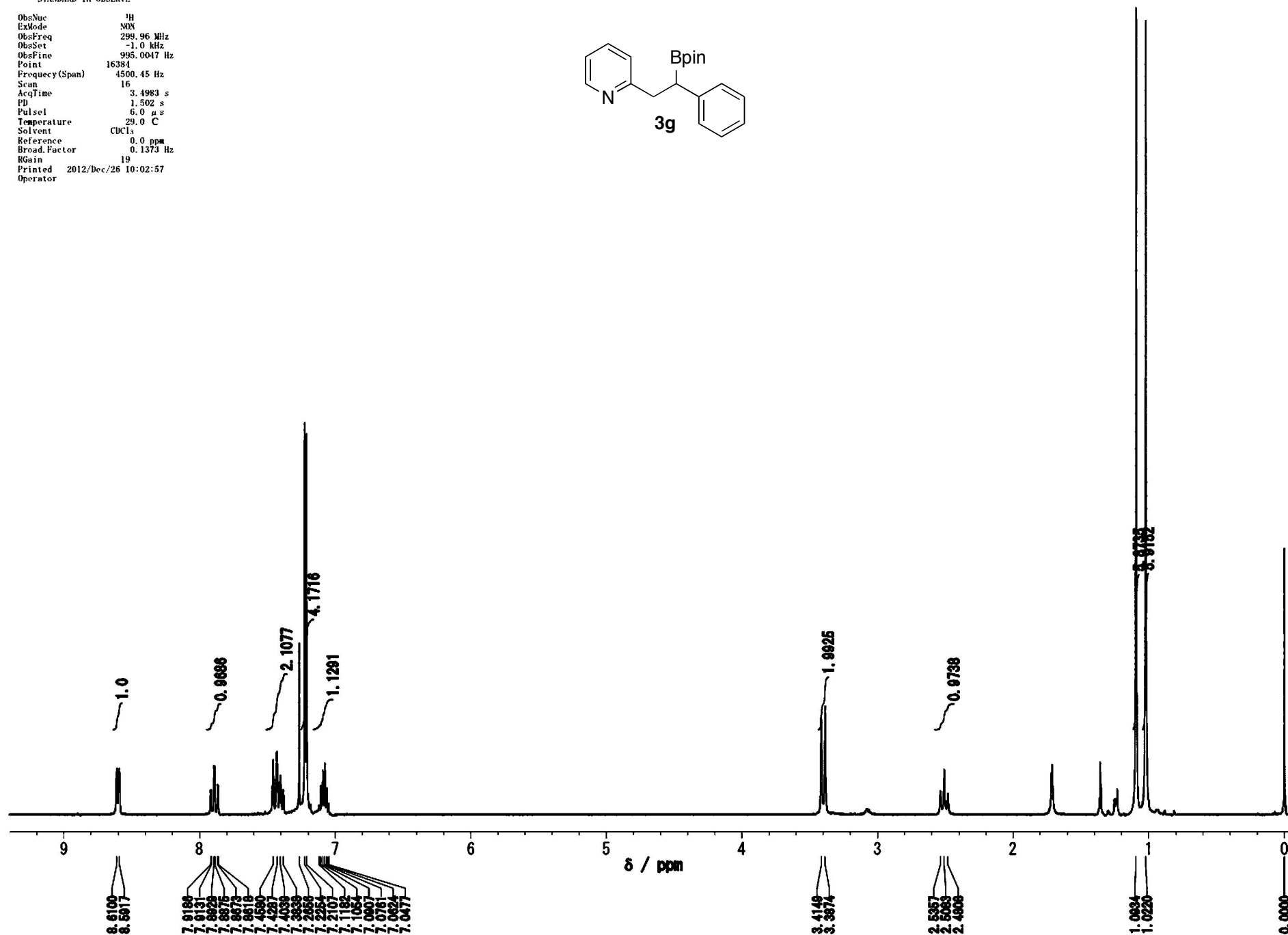
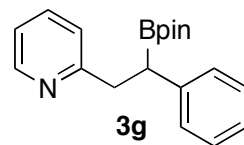
ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 09:46:48
 Operator



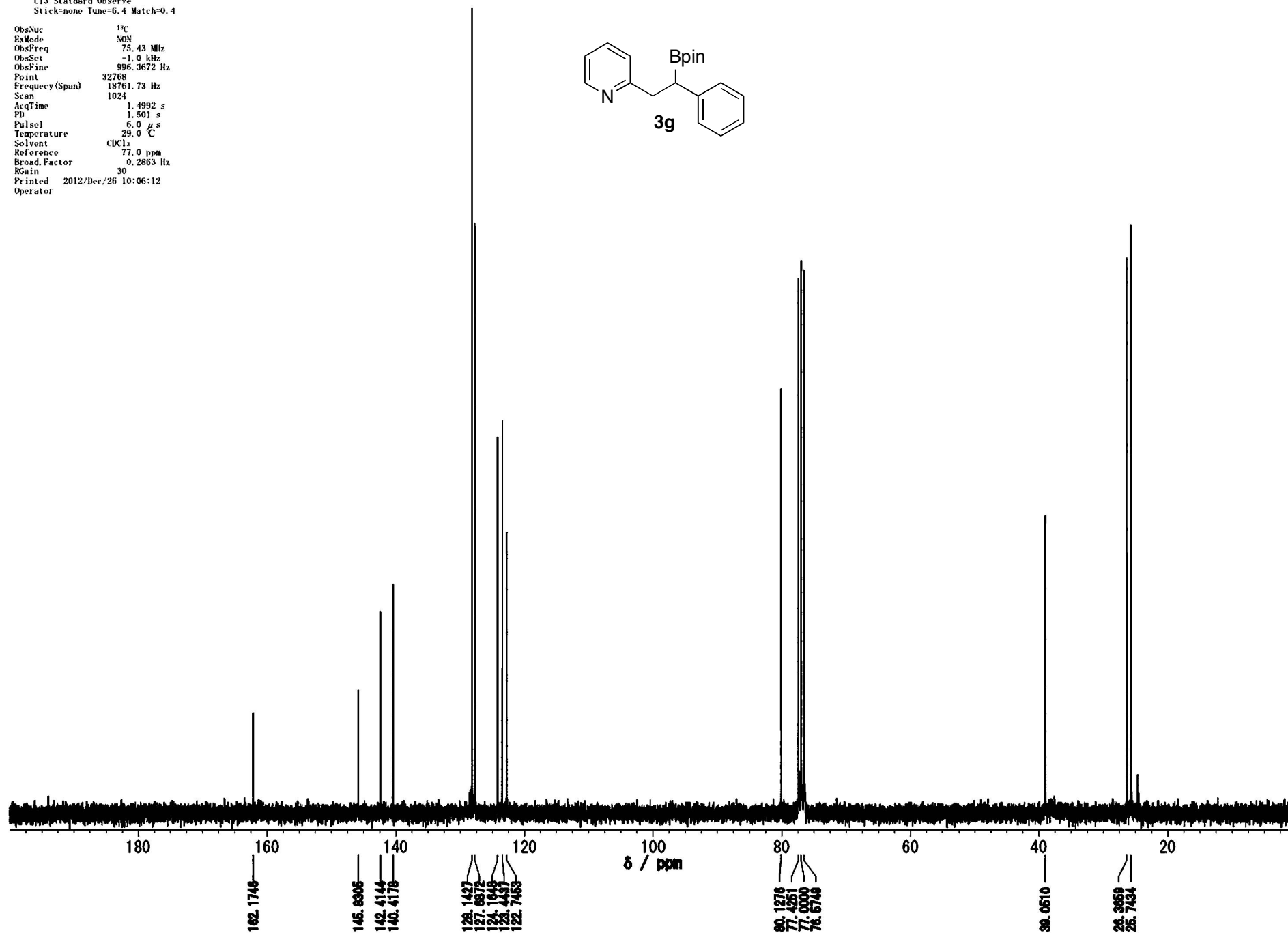
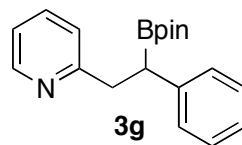
Date Oct 4 12

Comment
STANDARD 1H OBSERVE

ObsNuc ¹H
ExMode MON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsFine 995.0047 Hz
Point 16384
Frequency(Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μ s
Temperature 29.0 C
Solvent CDCl₃
Reference 0.0 ppm
Broad.Factor 0.1373 Hz
RGain 19
Printed 2012/Dec/26 10:02:57
Operator



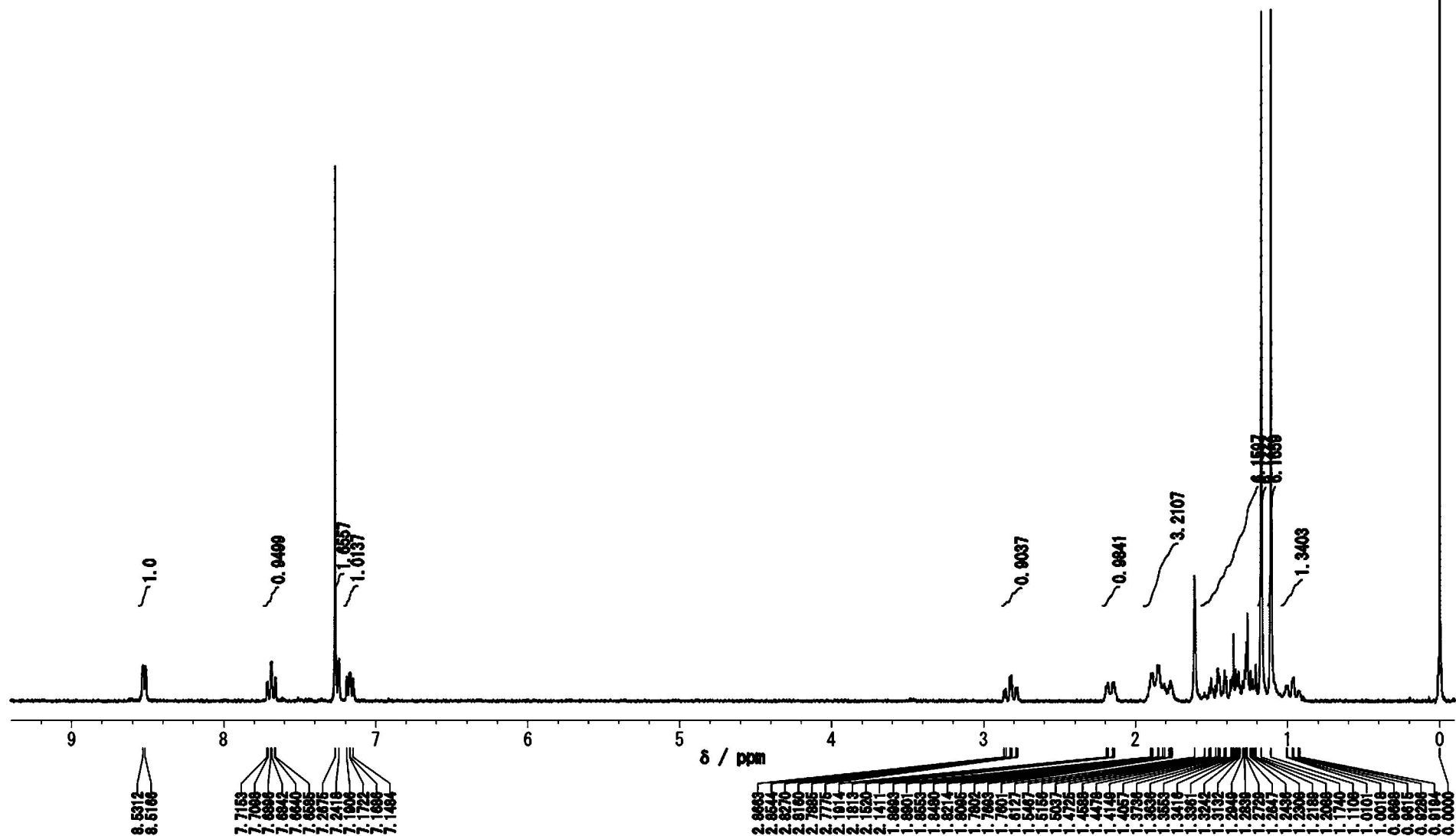
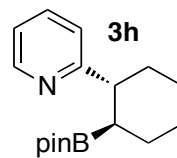
Date Nov 20 12
 Comment C13 Standard Observe
 Stick=none Tune=6.4 Match=0.4
 ObsNuc ^{13}C
 ExMode NOON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 995.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse1 6.0 μs
 Temperature 29.0 $^{\circ}\text{C}$
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 10:06:12
 Operator



Date Nov 16 12

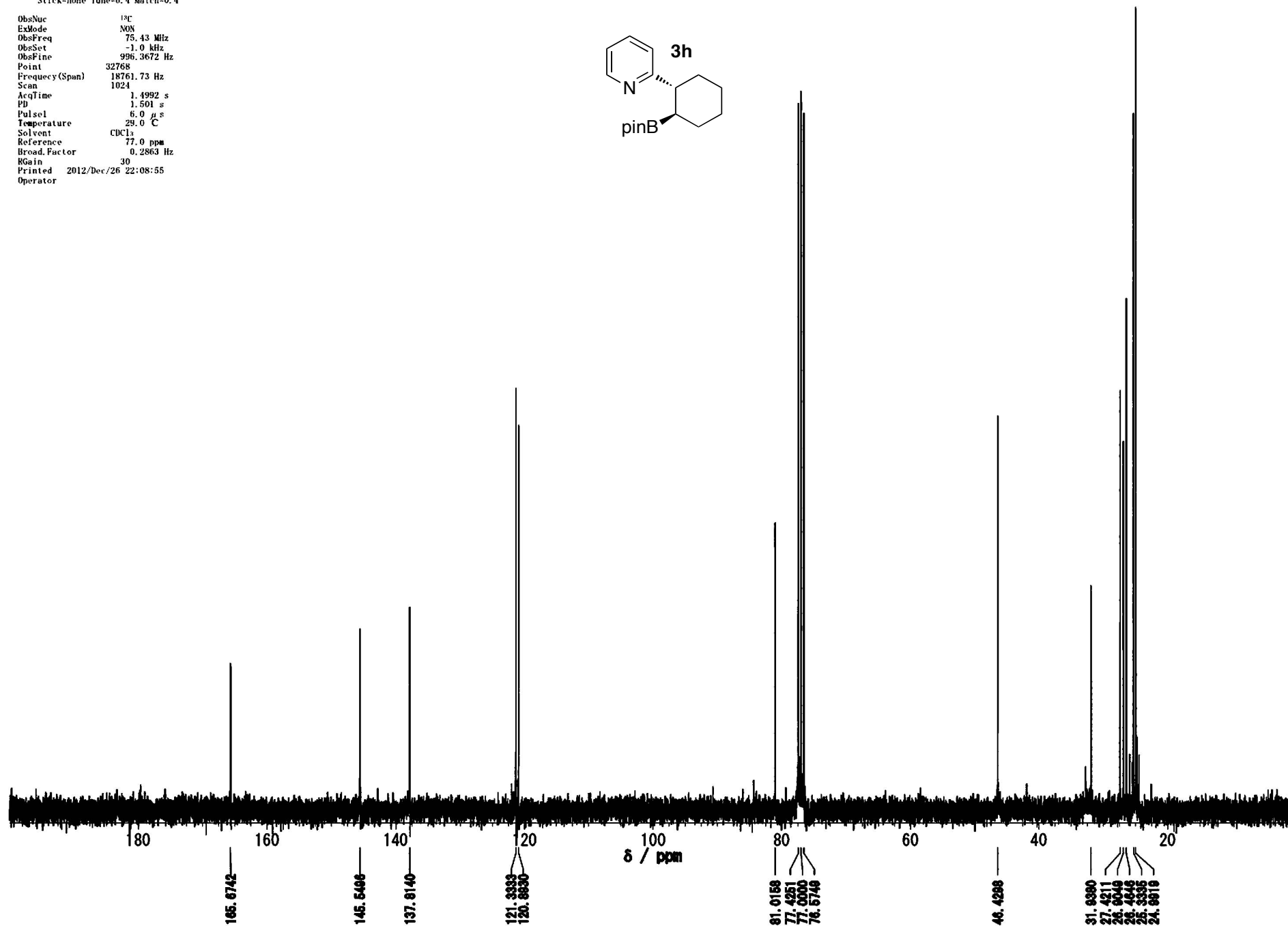
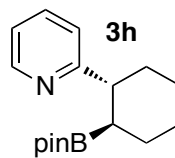
Comment
STANDARD 1H OBSERVE

ObsNuc 1H
ExMode NON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsFine 995.0047 Hz
Point 16384
Frequency (Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μs
Temperature 29.0 °C
Solvent CDCl₃
Reference 0.0 ppm
Broad Factor 0.1373 Hz
RGain 30
Printed 2012/Dec/26 09:43:06
Operator



Date Nov 19 12
 Comment C13 Standard Observe
 Stick=None Tune=6.4 Match=0.4

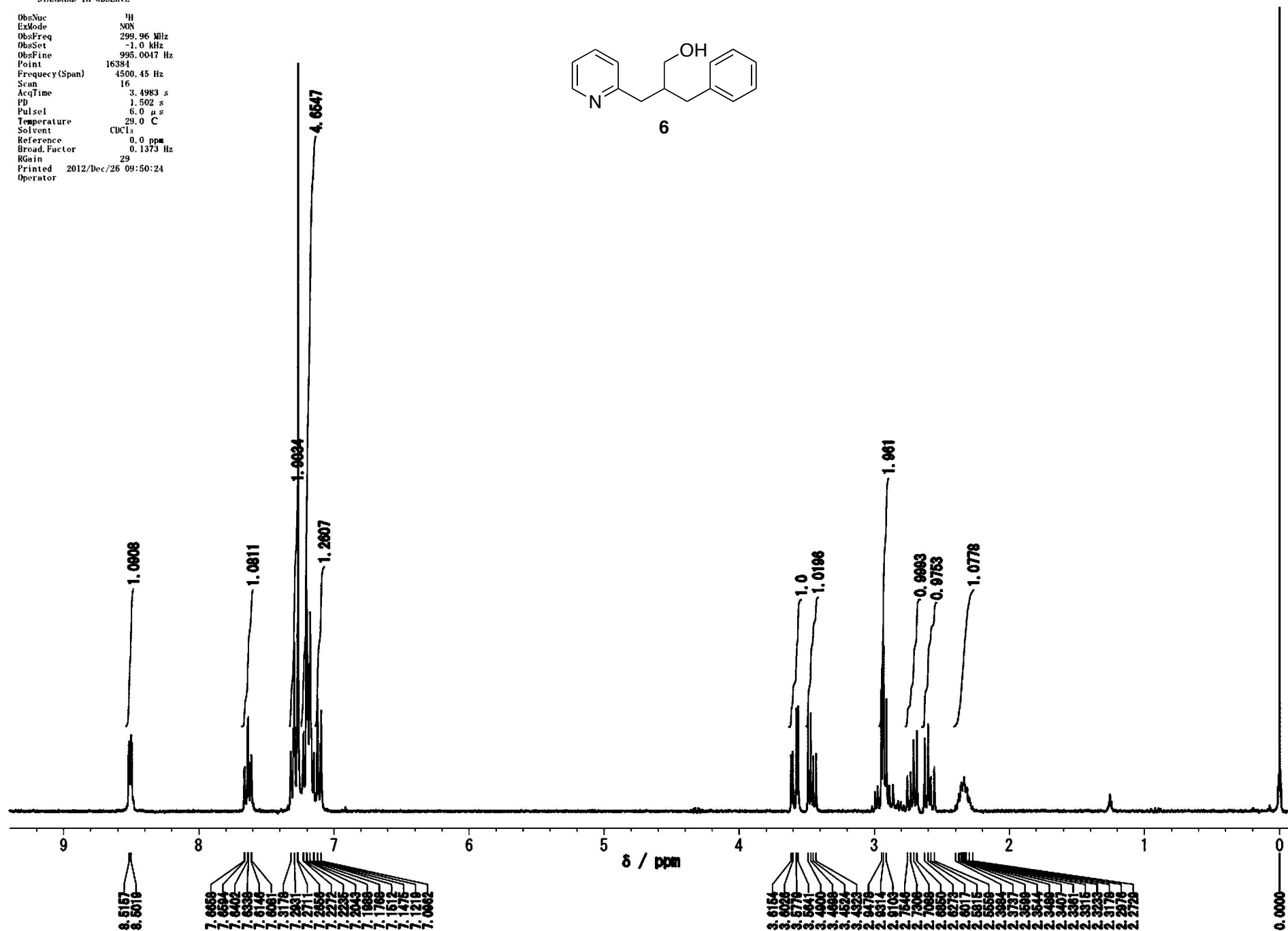
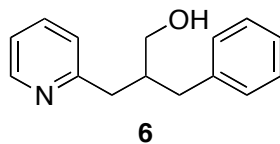
ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency(Span) 18761.73 Hz
 Scan 1024
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 22:08:55
 Operator



Date Dec 26 12

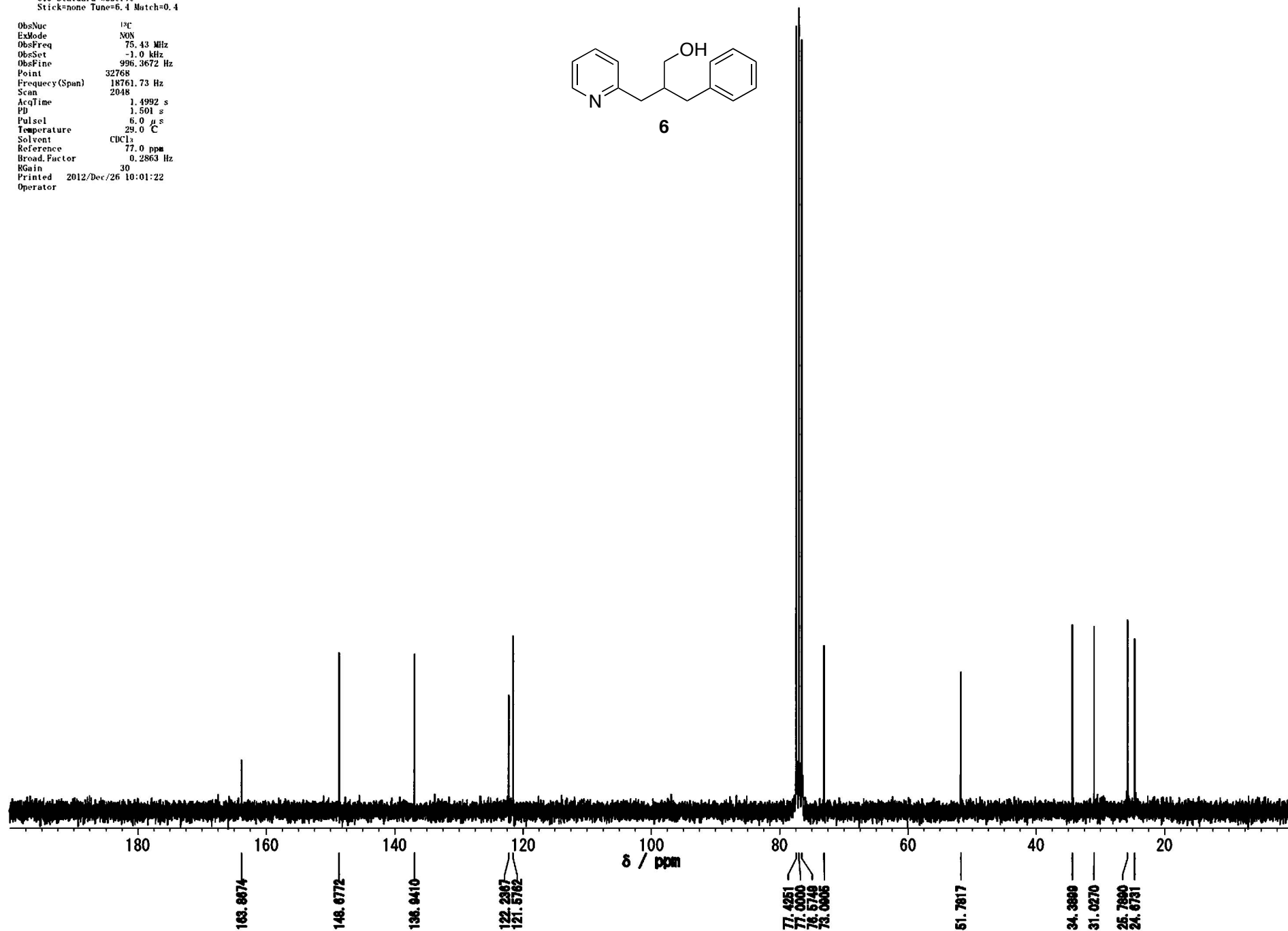
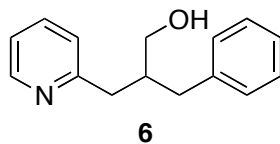
Comment
STANDARD 1H OBSERVE

ObsNuc ¹H
ExMode MON
ObsFreq 299.96 MHz
ObsSet -1.0 kHz
ObsFine 995.0047 Hz
Point 16384
Frequency(Span) 4500.45 Hz
Scan 16
AcqTime 3.4983 s
PD 1.502 s
Pulse 6.0 μ s
Temperature 29.0 C
Solvent CDCl₃
Reference 0.0 ppm
Broad.Factor 0.1373 Hz
RGain 29
Printed 2012/Dec/26 09:50:24
Operator



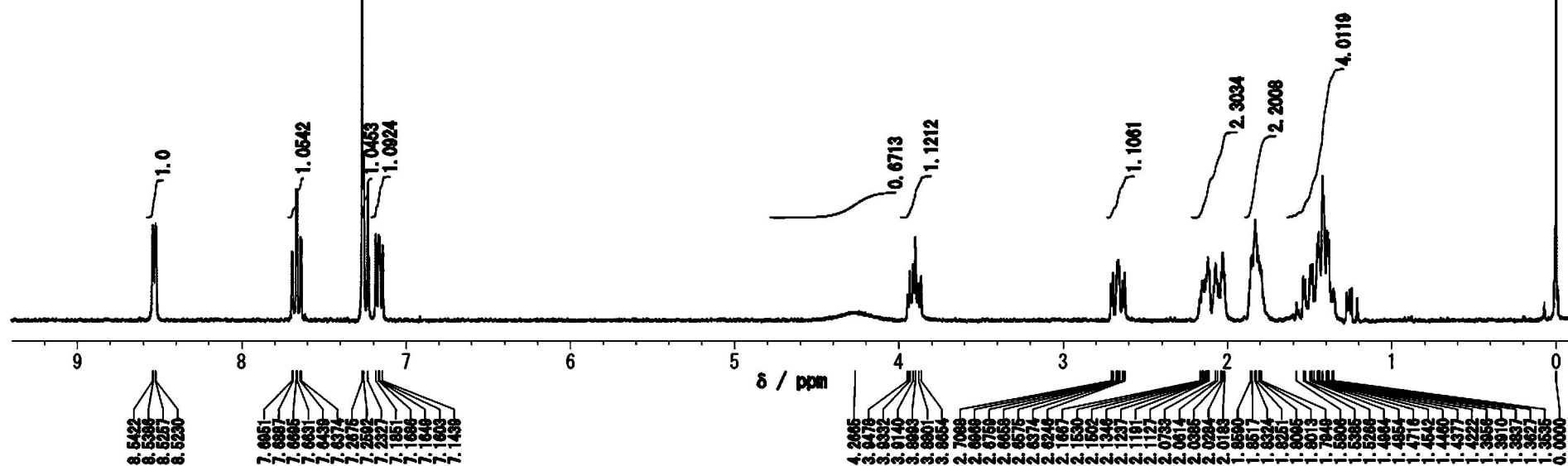
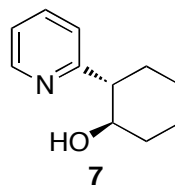
Date Dec 25 12
 Comment C13 Standard Observe
 Stick=None Tune=6.4 Match=0.4

ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSet -1.0 kHz
 ObsFine 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 2048
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μs
 Temperature 29.0 °C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad.Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 10:01:22
 Operator



Date Dec 18 12
 Comment STANDARD 1H OBSERVE

ObsNuc 1H
 ExMode NON
 ObsFreq 299.96 MHz
 ObsSet -1.0 kHz
 ObsFine 995.0047 Hz
 Point 16384
 Frequency (Span) 4500.45 Hz
 Scan 16
 AcqTime 3.4983 s
 PD 1.502 s
 Pulsol 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 0.0 ppm
 Broad Factor 0.1373 Hz
 RGain 30
 Printed 2012/Dec/26 09:59:51
 Operator



Date Dec 19 12
 Comment C13 Standard Observe
 Stick:none Tune=6.4 Match=0.4
 ObsNuc ¹³C
 ExMode NON
 ObsFreq 75.43 MHz
 ObsSct -1.9 kHz
 ObsFme 996.3672 Hz
 Point 32768
 Frequency (Span) 18761.73 Hz
 Scan 256
 AcqTime 1.4992 s
 PD 1.501 s
 Pulse 6.0 μ s
 Temperature 29.0 $^{\circ}$ C
 Solvent CDCl₃
 Reference 77.0 ppm
 Broad Factor 0.2863 Hz
 RGain 30
 Printed 2012/Dec/26 09:51:54
 Operator

